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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In Re Application of:

Kevan Hatchman, Elvin Lukenbach, Laura McCulloch, Benjamin Wiegand

Serial No.: 10/018,238

Group Art Unit: Not yet assigned

Filing Date: December 7, 2001

Examiner: Not yet assigned

For: PERSONAL CARE FORMULATIONS

DATE OF DEPOSIT:

April 9, 2002

I HEREBY CERTIFY THAT THIS PAPER IS BEING DEPOSITED WITH THE UNITED STATES POSTAL SERVICE AS FIRST CLASS MAIL, POSTAGE PREPAID ON THE DATE INDICATED ABOVE AND IS ADDRESSED TO ATTENTION: OFFICE OF PETITIONS, BOX DAC, ASSISTANT COMMISSIONER FOR PATENTS, WASHINGTON, DC 20231.

Wendy A. Choi  
Wendy A. Choi, Esquire  
REGISTRATION NO.: 36,697

BOX DAC

Office of Petitions

Assistant Commissioner for Patents

Washington, DC 20231

### PETITION FOR RETROACTIVE LICENSE UNDER 37 CFR 5.25

It is respectfully requested that this petition for license for foreign filing attached hereto be granted retroactively under the provisions of 37 CFR 5.25.

Previous Licenses ( ☐ applicable ☒ not applicable)

Attached are copies of:



previous licenses



the filing receipt license

issued on this invention before the export.

### Material filed abroad without a license

Attached is a copy of the material that was filed abroad without a license for foreign filing.

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**Foreign Countries and dates of filing of material for which retroactive license requested per 37 CFR 5.25(a)(1).**

With respect to the material for which a retroactive license is requested, each foreign country in which the patent application material was filed and the date it was filed is as follows:

<b>Foreign Country</b>	<b>Date</b>	<b>Application No.</b>
<u>Great Britain (UK Patent Office)</u>	<u>10 June 1999</u>	<u>GB 99 13408.2</u>
<u>Germany (EPO PCT Receiving Office)</u>	<u>9 June 2000</u>	<u>WO 00/76460</u>
<u>Germany (European Patent Office)</u>	<u>5 December 2001</u>	<u>EP 00945731.8</u>
<u>Australia</u>	<u>7 December 2001</u>	<u>ASL 59716/00</u>

**Verified Statement(s)**

Also attached hereto are the verified statement(s) (oath or declaration) of:

Wendy A. Choi, Esq. attorney for Johnson & Johnson Consumer Companies, Inc.,  
joins assignee of 10/018,238

which confirm(s) that, in accordance with 37 CFR 5.25(a)(3)(I)-(iii),

(a) the subject matter in question was not under a secrecy order at the time it was filed abroad, and that it is not currently under a secrecy order;

(b) the license is being diligently sought after discovery of the proscribed foreign filing; and

(c) an explanation of why the material was filed abroad through error and without deceptive intent without the required license under §5.11 first having been obtained.

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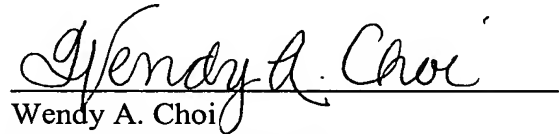
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Fee under 37 CFR 1.17(h) \$130.00

☒ A Check is Enclosed in the Foregoing Amount Due.

☒ The Commissioner is further authorized to charge any fees related to any such extension of time to deposit account 23-3050. This sheet is provided in duplicate.

Date: April 9, 2002

  
Wendy A. Choi  
Registration No. 36,697

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Philadelphia PA 19103  
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re patent application of :

Kevin Hatchman, Elvin Lukenbach, Lachar  
McCulloch and Benjamin Wiegand.

Serial No. : 10/018,238

Group No. : Not yet assigned

Filed : 12/07/01

Examiner : Not yet assigned

For : PERSONAL CARE FORMULATIONS

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Office of Petitions

Assistant Commissioner for Patents

Washington, D.C. 20231

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Dear Sir:

**STATEMENT OF FACTS REGARDING FAILURE TO SEEK U.S. FOREIGN  
FILING LICENSE UNDER 35 U.S.C § 184**

Applicants respectfully request a retroactively granted license under 35 U.S.C. § 1811 to the subject matter of GB 9913408.2, WO 00/76460 and related national stage applications, which were filed outside of the U.S. without a prior license from the Commissioner of Patents and Trademarks through error and without deceptive intent.

***Summary of Facts***

The subject matter of GB 9913408.2, WO 00/76460 and related national stage applications is directed to personal care formulations, such as shampoo and body wash. More specifically, the subject matter relates to clear, gel-like personal care formulations that contain surfactant, oil and water. The oil may be a mineral oil, fatty ester, glyceride, terpene or silicone oil and the surfactant has an HLB of 2-10.

The technology that is the subject-matter of the applications was jointly developed by Albright & Wilson Surfactants, a subsidiary of Rhodia Consumer Specialities, Ltd. (A&W) and Johnson & Johnson Consumer Companies, Inc. (J&J). A&W is located in the U.K. and develops and manufactures gels. J&J is located in the U.S. and develops, manufactures and sells hair and skin care products. The two parties were working together initially under a secrecy agreement and eventually under a joint development agreement to develop suitable A&W gels for J&J's hair and skin care products.

During a teleconference between personnel of both parties on June 6, 1999, J&J was informed by A&W that A&W intended to file a provisional British patent application directed to an invention made solely by personnel of A&W. Without

review by any personnel at J&J, this application (GB 9913408.2) was filed on June 10, 1999 by Mr. Roger Savidge, a patent attorney for Rhodia. As a provisional British patent application, it did not name any inventors (only A&W as the applicant). A copy of the application, as filed, was faxed to J&J on June 10, 1999 (*Exhibit 1*). After reviewing the patent application, J&J determined that J&J personnel (who made the invention in the U.S.) were at least joint inventors on the provisional British patent application. Through ignorance of the requirement, neither party realized that, because the invention disclosed in the British application was at least partially made in the U.S., that it was necessary to seek a foreign filing license in the U.S. prior to filing the British application.

On the eve of the Paris Convention deadline of June 9, 2000, Mr. Savidge contacted Ms. Michele Mangini, a patent attorney with J&J, to discuss filing a PCT application based upon the provisional British application by June 9, 2000. On June 8, 2000, Ms. Mangini signed, as Assistant Secretary of J&J, and returned via facsimile a document appointing Mr. Savidge as the representative for the PCT application (*Exhibit 2*).

The PCT application was filed on June 9, 2000 (WO 00/76460)(*Exhibit 3*). There were four inventors listed on the PCT application:

- (1) Kevan HATCHMAN (A&W)
- (2) Elvin LUKENBACH (J&J)
- (3) Laura McCULLOCH (J&J)
- (4) Benjamin WIEGAND (J&J)

Inventors (2)-(4) made at least a part of their contribution to the claimed invention in the U.S. and while residing in the U.S. Mr. Savidge handled the prosecution of the PCT application with input from J&J. The PCT application is essentially identical to the provisional British application. In haste to secure a filing date and again through continuing ignorance of the requirement, neither party realized that, because the invention disclosed in the British and PCT applications was at least partially made in the U.S., that it was necessary to seek a foreign filing license in the U.S. prior to filing the PCT application and seek a retroactive license with respect to the provisional British application.

At the 30-month deadline for entering Chapter II of the PCT, Mr. Savidge engaged a U.S. patent attorney, Mr. Marshall Chick of FRISHAUF, HOLTZ, GOODMAN, LANGER & CHICK, to file a U.S. national application (SN 10/018,238 on December 7, 2001) based on the PCT application. National applications were also filed at the EPO and in Australia, based on instructions from Mr. Savidge (*Exhibit 4* and *Exhibit 5*, respectively). Instructions were sent to a Canadian agent to file a Canadian national application. However, due to conflicting instructions by J&J, the Canadian agent did not file (and has not yet filed) the application in Canada (*Exhibit 6*). Applicants intend to file the application in Canada, provided that and only after the retroactive license requested herein is granted.

On or about January 15, 2002, Mr. Chick contacted J&J to collect details to prepare a post-filed declaration, power of attorney and assignment (*Exhibit 7*). In reviewing the application papers from Mr. Savidge, Mr. Chick discovered that there was no record of the granting of a U.S. foreign filing license. He inquired whether

J&J had sought a U.S. foreign filing license and could not confirm that either party had sought such a license.

On February 6, 2002, Mr. Chick forwarded the U.S. application to me (*Exhibit 8*), Wendy Choi of WOODCOCK WASHBURN LLP, a law firm now representing J&J, to handle the prosecution of the application, including seeking a retroactive foreign filing license.

I have reviewed all of the relevant documents and discussed the history of the filings with Ms. Mangini, Mr. Savidge and Dr. Wiegand. As a result of my review, I have prepared this statement of facts to support the petition for a retroactively granted license under 37 C.F.R. § 5.25.

### **Conclusion**

Applicants respectfully request a retroactively granted license under 35 U.S.C. § 184 and 37 C.F.R. § 5.25 to the subject matter of GB 9913408.2 and WO 00/76460 and related national stage applications, which were filed outside of the U.S. without a license from the Commissioner of Patents and Trademarks. Applicants submit that:

- (1) the applications that were filed without the required licenses under 35 U.S.C. § 184 were *filed through error and without deceptive intent*;
- (2) the subject matter of the applications was not under a secrecy order under 35 U.S.C. § 181 at the time that the applications was filed abroad, and the subject matter is not currently under a secrecy order; and
- (3) they are promptly seeking a retroactive license under 35 U.S.C. § 184 and 37 C.F.R. § 5.25 after discovery of their error in not seeking the required license.

The required fee in accordance with 37 C.F.R. § 1.17(h) is enclosed.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under 18 U.S.C. § 1001.

Respectfully submitted,



Wendy A. Choi  
Registration No. 36,697  
Attorney for Applicants

Date: April 9, 2002  
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# Exhibit 1

# fax message

From R G M Savidge  
Corporate Patents Mgr

Tel 0121 420 5430

Fax 0121 420 5437

To Ben Wiegand

Org Johnson & Johnson

Fax 001 908 874 1126

Page 4 of 32

Date June 10, 1999

Ref MPL315/GB/RGMS  
PBF1

CC

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GB13408.2

filed 06/10/99

British Provisional  
Priority Document

Dear Ben

### RINGING GEL TECHNOLOGY

As promised I enclose a copy of our informal preliminary patent application which was filed at the UK Patent Office today in order to secure a priority date for a future substantive patent application to be filed within the next 12 months. This application does not specify the inventorship.

Please let me have any comments or suggested additions or modifications. If there are any points of substance I will file a further improved text.

I also enclose a copy of our EP O 598 335 which was filed six years ago and then abandoned. Our corresponding Canadian application may still be revivable until November of this year.

Regards.

*Rob*

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DEC 15 1999

MICHELE G. MANGINI

Encs

Albright & Wilson UK Limited  
Place of Registration: England  
Registered Number 36833  
Registered Office:  
213-222 Hagley Road West  
Oldbury  
West Midlands B68 8NN



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## PERSONAL CARE FORMULATIONS

The present invention relates to shampoo or cleaning compositions suitable for personal care applications in the form of  $I_1$  mesophase systems containing dispersed oil.

Dispersing oil in aqueous shampoo and body wash formulations has presented problems. To prevent the oil phase separating it must either be: (A) emulsified which involves dispersing the oil as colloidal single droplets; (B) microemulsified which involves forming a micellar solution with oil incorporated into surfactant micelles; (C) suspended in a structured surfactant system which typically comprises a dispersion of a surfactant mesophase in aqueous electrolyte; or (D) incorporated into a water soluble solid, pasty or gelatinous composition.

With the exception of microemulsions which are clear, thermodynamically stable, micellar solutions, the foregoing systems are necessarily opaque and contain the oil dispersed in a relatively coarse form, which does not deposit satisfactorily on skin or hair.

However microemulsions are difficult to formulate using the surfactants which are most effective in body wash and other personal care formulations and contain relatively low concentrations of surfactant.

We have now discovered that oil may be stably incorporated into the structure of an  $I_1$  phase to form a clear gel-like composition which contains higher concentrations of surfactant and oil than conventional microemulsions, but, which dissolves in water to form a microemulsion. The novel oil-in- $I_1$  compositions also form microemulsions on heating.

Surfactants are known to form mesophases or liquid crystal phases at concentrations above approximately 30% by weight based on the weight of water and surfactant. Mesophases are phases which exhibit a degree of order intermediate between typical liquids and solids. Generally mesophases combine long range order associated with crystals, with fast molecular motion common to liquids.

The formation of detergent mesophases is well documented. Different surfactants and surfactant mixtures differ widely in their ability to form the numerous different mesophases, and in respect of the conditions of concentration and temperature at which they are formed. For a typical surfactant of the type normally used in cleaning products the following mesophases are usually observed. The concentrations given are illustrative only and may vary considerably from one surfactant or surfactant mixture to the next.

Below approximately 30% surfactant an isotropic  $L_1$  phase is formed (with micelles of surfactant in water). Above 30% surfactant many detergents form a M phase which is not normally used in personal care applications since it does not show suitable flow characteristics and is difficult to dissolve or disperse in water. Above the concentrations required to form an M phase, but usually at concentrations of less than 80% active surfactant, i.e. 60%-80% a G-phase is formed. At concentrations higher than those required to form a G-phase, i.e. typically greater than 80% active surfactant, most surfactants form a hydrated solid, and some, especially non-ionic surfactants form a liquid phase containing dispersed micelle sized droplets of water - an inverted micellar solution known as an  $L_2$  phase.  $L_2$  detergent systems do not disperse readily in water and have a tendency to form undesirable gels, e.g. M phases, on dilution.

Some surfactants form viscous isotropic or VI phases. These are immobile phases usually with a vitreous appearance, and have been relatively little studied compared to the other phases discussed above. They have been virtually ignored in the context of formulating cleaning compositions because most of the surfactants and surfactant systems which are commonly used in cleaning compositions do not form VI phases, at least at normal temperatures, or form them only within narrow concentration ranges and because their known properties as immobile gels has deterred formulators from investigating them. They are recognised as being the most viscous of the lyotropic mesophases.

The different surfactant phases can be recognised by a combination of appearance, rheology, textures under the microscope, electron microscopy and x-ray diffraction or neutron scattering. A detailed description, with illustrations, of the difference textures observable using a polarising microscope, is to be found in the paper by Rosevear JAOCS Vol 31, p628.

The following terms may require explanation or definition:

The "hydrophilic: lipophilic balance", or "HLB" value is used as a measure of the relative affinities of the surfactants for water and oil respectively and correlates with their effectiveness as emulsifiers. HLB value can easily be calculated for alcohol ethoxylates since it is one fifth of the weight percent of ethylene oxide based on the total mole weight. Other surfactants can be assigned equivalent values by applying more complicated formulae or by measuring their relative affinity for water and oil. An HLB value of 20 represents a completely water soluble oil insoluble surfactant, while an HLB value of 0 represents a completely oil soluble and water insoluble surfactant.



"Optically isotropic" surfactant phases do not normally tend to rotate the plane of polarisation of plane polarised light. If a drop of sample is placed between two sheets of optically plane polarising material whose planes are at right angles, and light is shone on to one sheet, optically isotropic surfactant samples do not appear substantially brighter than their surrounding when viewed through the other sheet. Optically anisotropic materials appear substantially brighter. Optically anisotropic mesophases typically show characteristic textures when viewed through a microscope between crossed polarisers, whereas optically isotropic phases usually show a featureless continuum.

"Newtonian liquids" have a viscosity which remains constant at different shear rates. For the purpose of this specification, liquids are considered Newtonian if the viscosity does not vary substantially at shear rates up to  $1000 \text{ sec}^{-1}$ .

"Lamellar" phases are phases which comprise a plurality of bilayers of surfactant arranged in parallel and separated by liquid medium. They include both solid phases and the typical form of the liquid crystal G-phase. G-phases are typically pourable, non-Newtonian, anisotropic products. They are typically viscous-looking, opalescent materials with a characteristic "smeary" appearance on flowing. They form characteristic texture under the polarising microscope and freeze fractured samples have a lamellar appearance under the electron microscope. X-ray diffraction or neutron scattering similarly reveal a lamellar structure, with a principal peak typically between 4 and 10nm, usually 5 to 6nm. Higher order peaks, when present occur at double or higher integral multiples of the Q value of the principal peak. Q is the momentum transfer vector and is related, in the case of lamellar phases, to the repeat spacing d by the equation  $Q = \frac{2\pi}{d} [\text{Å}^{-1}]$  where n is the order of the peak.

G-phases, however, can exist in several different forms, including domains of parallel sheets which constitute the bulk of the typical G-phases described above and spherulites formed from a number of concentric spheroidal shells, each of which is a bilayer of surfactant. In this specification the term "lamellar" will be reserved for compositions which are at least partly of the former type. Opaque compositions at least predominantly of the latter type in which the continuous phase is a substantially isotropic solution containing dispersed spherulites are referred to herein as "G-phase compositions". G-phases are sometimes referred to in the literature as  $L_{(1)pha}$  phases.

$L_1$ -phases are mobile, optically isotropic, and typically Newtonian liquids which show no texture under the polarising microscope. Electron microscopy is capable of resolving the texture of such phases only at very high magnifications, and X-ray or neutron scattering normally gives only a single broad peak typical of a liquid structure, at very small angles close to the reference beam. The viscosity of an  $L_1$ -phase is usually low, but may rise significantly as the concentration approaches the upper phase boundary.

"M-phases" are typically immobile, anisotropic products resembling low melting point waxes. They give characteristic textures under the polarising microscope, and a hexagonal diffraction pattern by X-ray or neutron diffraction which comprises a major peak, usually at values corresponding to a repeat spacing between 4 and 10nm, and sometimes higher order peaks, the first at a Q-value which is  $3^{0.5}$  times the Q-value of the principal peak and the next double the Q-value of the principal peak. M-phases are sometimes referred to in the literature as H-phases.

The viscous isotropic or "VI" phases are typically immobile, non-Newtonian, optically isotropic and are typically transparent, at least when pure. VI phases have a cubic symmetrical diffraction pattern, under X-ray diffraction or neutron scattering, with a principal peak and higher order peaks at  $2^{0.5}$  and  $3^{0.5}$  times the Q-value of the principal peak.

These cubic liquid crystalline phases are sometimes observed immediately following the uniceilar phase at ambient temperature as the concentration of surfactant is increased. It has been proposed that such VI phases, sometimes referred to as  $I_1$  phase, may arise from the packing of micelles (probably spherical) in a cubic lattice. At ambient temperature a further increase in surfactant concentration usually results in hexagonal phase ( $M_1$ ), which may be followed by a lamellar phase (G).  $I_1$  phases, when they occur, are usually only observed over a narrow range of concentrations, typically just above those at which the  $L_1$ -phase is formed. The location of such VI phases in a phase diagram suggests that the phase is built up of small closed surfactant aggregates in a water continuum.

An inverse form of the  $I_1$  phase (the  $I_2$  phase) has also been reported, possibly between the inverse hexagonal ( $M_2$ ) and  $L_2$  phases. It consists of a surfactant continuum containing a cubic array of inverted micelles. An alternative form of the VI phase called the  $V_1$  phase has been observed at concentrations between the M and G phases and may comprise a bicontinuous system. This may exhibit an even higher viscosity than the  $I_1$ . An inverse phase, the  $V_2$  phase, between the G and  $M_2$  phases has also been postulated.

VI phases are typically examples of "ringing gels". When a jar or beaker containing such a phase is sharply struck, a distinctive vibration can be felt in the composition.

The  $I_1/L_1$  transition temperature will be referred to herein as the melting point of the  $I_1$  phase for convenience, although it is not strictly speaking the melting point since the VI phases are not solids.

All references herein to the formation or existence of specific phases or structures are to be construed, unless the context requires otherwise, as references to their formation or existence at 20°C.

Hexagonal gels (M-phase) have been referred to in the prior art as cleaning compositions, e.g. GB 2 179 055, EP 1 153 837 and colloidal gels formed with gelling agents such as synthetic polymers or gelatin have also been suggested, e.g. US 4 465 663.

However these compositions cannot be readily dissolved in water to form microemulsions. They are moreover usually opaque and of an unattractive appearance and often require the presence of solvents such as glycols which add to the cost and are environmentally undesirable.

The use of a type of ringing gel to suspend oil for cosmetic or pharmaceutical applications was described in US 4 026 318 but the formulation requires the presence of hydroxylic solvents and utilises a surfactant system which is unsuitable for shampoo applications. EP 0 598 335 describes the use of various cubic phases including  $I_1$  phases as laundry prespotters and for other cleaning formulations. It does not suggest how such phases could be used to suspend oil or form microemulsions. Normally attempts to suspend oil in surfactant mesophases result in coarse droplets of oil being suspended in the aqueous phase of a structured surfactant.

Our invention provides a concentrated personal cleansing composition comprising at least 20% water, 10 to 40% total surfactant and 2 to 40% of a mineral, glyceride, terpene or silicone oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.

The surfactants are preferably selected to provide an  $I_1$  phase over a comparatively broad surfactant concentration range e.g. more than  $\pm 5\%$  or greater, which range typically lies above 15% by weight total surfactant based on the weight of the composition e.g. between 20% and 40% by weight surfactant usually between 25% and 60%.

The surfactants are preferably selected to provide an  $I_1$  phase which melts above 30°C e.g. above 35°C, most preferably above 40°C. Preferably the  $I_1$  phase melts at a temperature substantially below 100°C, e.g. below 90°C, more preferably below 80°C, most preferably below 70°C, especially below 60°C, typically below 55°C, usually below 50°C.

The surfactant mixture preferably has a mean HLB based on the molar proportions of the components between 10 and 15 e.g. 11 to 14. The surfactants preferably comprise non-ionic surfactants such as ethoxylated alcohols. It has been found that highly ethoxylated fatty alcohols, e.g. more than 10 EO groups, preferably more than 15 EO groups, especially 18 to 50 EO groups form  $I_1$  phases particularly readily.

Other non-ionic surfactants which may be present include:-

alkyl phenol ethoxylates, fatty acid ethoxylates, fatty acid monoalkylolamide ethoxylates, fatty alcohol propoxylates, fatty amine alkoxyates and fatty acid glyceryl ester ethoxylates. Other non-ionic compounds suitable for inclusion in compositions of the present invention include mixed ethylene oxide propylene oxide alkoxyates, low relative molecular mass polyethylene glycols e.g. PEG600 and PEG200, ethylene glycol monoesters, amine oxides and alkyl polyglycosides, alkyl sugar esters including alkyl sucrose esters and alkyl oligosaccharide ester, alkyl capped polyvinyl alcohol and alkyl capped polyvinyl pyrrolidone.

Compositions of the invention may also comprise anionic surfactants, in addition to or instead of non-ionic surfactants. Anionic surfactant may comprise a  $C_{10-20}$  alkyl benzene sulphonate or an alkyl ether sulphate which is preferably the product obtained by ethoxylating a natural fatty or synthetic  $C_{10-20}$  e.g. a  $C_{12-14}$  alcohol with from 1 to 20, preferably 2 to 10 e.g. 3 to 4 ethyleneoxy groups, optionally stripping any unreacted alcohol, reacting the ethoxylated product with a sulphating agent and neutralising the resulting alkyl ether sulphuric acid with a base. The term also includes alkyl glyceryl sulphates, and random or block copolymerised alkyl ethoxypropoxy sulphates.

The anionic surfactant may also comprise, for example,  $C_{10-20}$  e.g.  $C_{12-14}$  alkyl sulphate.

The surfactant may comprise a  $C_{10-20}$  e.g.  $C_{12-14}$  aliphatic soap. The soap may be saturated or unsaturated, straight or branched chain.

Preferred examples include dodecanoates, myristates, stearates, oleates, linoleates, linoleates and palmitates and coconut and tallow soaps.

The surfactant may include other anionic surfactants, such as olefin sulphonates, paraffin sulphonates, taurides, isethionates, ether sulphonates, ether carboxylates, aliphatic ester sulphonates e.g. alkyl glyceryl sulphonates, sulphosuccinates or sulphosuccinamates.

The cation of any anionic surfactant is typically sodium but may alternatively be potassium, lithium, calcium, magnesium, ammonium, or an alkyl ammonium having up to 6 aliphatic carbon atoms including isopropyl ammonium, monoethanol ammonium, diethanol ammonium, and triethanol ammonium.

Ammonium and ethanol ammonium salts are generally more soluble than the sodium salts. Mixtures of the above cations may be used.

The composition may contain amphoteric surfactants such as betaines sulphobetaines, amido betaines or imidazoline betaines.

The  $I_1$  phase may be conveniently prepared by mixing the oil and oil soluble surfactant and adding sufficient water to the water soluble surfactant to maintain a lamellar phase. The oil and oil soluble surfactant may be stirred into the lamellar composition at elevated temperature, above the melting point of the desired  $I_1$  phase. The composition is then diluted with hot water until a microemulsion is formed and then cooled to solidify it into the  $I_1$  phase.

The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether) or a fatty glyceride, a terpene oil such as limonene or a silicone oil. Mixtures of oils may be used. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil. Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolved in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds, ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide may be dispersed in the hot microemulsion prior to cooling. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may be suspended. Other active ingredients which may be suspended include insect repellants and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

Electrolytes tend to break  $L_1$  phase structure and are preferably present in concentrations below 10% based on total weight of the compositions, more preferably below 5%, e.g. 0 to 3%, most preferably 0 to 1%. Generally we prefer that electrolyte be substantially absent. Adventitious chloride or sulphate present as impurities in the surfactant can be tolerated. Small amounts of builder such as citrates, pyrophosphates, polyphosphates may optionally be included.

Water soluble solvents are generally undesirable and are not required to form stable  $L_1$  structures according to the invention. We therefore prefer that they should be substantially absent. Although small amounts of, for example, ethanol or propanol may sometimes be desired for special purposes, they are preferably present in amounts less than 5% by weight, more preferably less than 3% by weight, most preferably less than 2% by weight, e.g. less than 1% by weight.

The composition may optionally contain hydrotropes such as sodium lower alkyl benzene sulphonate e.g. sodium toluene, xylene or cumene sulphonate or urea, however these are not generally necessary and are not generally preferred. We prefer that these should be present in quantities less than 5% by weight, more preferably less than 4%, especially less than 2% e.g. 0 to 1%. They may be useful occasionally to avoid haziness of the gel.

The total amount of water is preferably from 25 to 60% by weight of the composition, more preferably 30 to 50%, e.g. 35 to 50%. The total weight percentage of surfactant based on the weight of the composition is preferably from 15 to 35%, e.g. 20 to 30%. The proportion of oil is preferably greater than 5%, more preferably greater than 8%, e.g. 10 to 30%, especially 15 to 25% by weight based on the weight of the composition. The oil soluble surfactant is preferably present in a proportion of more than 1.5 based on the weight of oil, more preferably from 1:2 to 5:1. The oil soluble surfactant preferably has an HLB of from 3 to 9 e.g. 4 to 8.



The weight ratio of water soluble surfactant to oil soluble surfactant is preferably 1:1 to 30:1, more preferably 2:1 to 20:1, typically 3:1 to 15:1, e.g. 4:1 to 10:1. The water soluble surfactant preferably has an HLB greater than 12, more preferably greater than 13, especially 14 to 19.

The product may be cast into shaped bodies or formed into particles or granules, e.g. by spray cooling a hot solution of the  $L_1$  phase formed on melting the composition.

The composition may be converted into a microemulsion phase by addition of water, by heating above the melting point or by adding electrolyte such as salt and the invention includes  $L_1$  phases when so prepared.

The invention will be illustrated by the following examples:

#### Example 1

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	20	20
"EMPICOL"® 0251/70J (70%)	11.2	16
"EMPIGEN"® BB (30%)	4.8	16
"GLUCAPON"® 215 CS UP (55%)	6	9.2
"EMPILAN"® KB2 (100%)	7.5	7.5
SODIUM CHLORIDE (100%)	2	2
PERFUME (100%)	0.5	0.5
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.1	0.1
CITRIC ACID (100%)	0.2	0.2
BENZOIC ACID (100%)	0.3	0.3
SODIUM HYDROXIDE (47%)	0.1	0.2
WATER	---	Balance

The method of mixing comprised the following steps:-

1. Charge 50% of water
2. Heat to 60°C
3. Add EDTA, sodium benzoate, citric acid and 47% NaOH dissolve with stirring
4. Add "EMPIGEN" BB
5. Add mineral oil and disperse with stirring
6. Add "EMPILAN" KB 2 and mix thoroughly
7. Add "EMPICOL" 0251/70j
8. Add remaining water
9. Add "GLUCAPON" 215 CS UP
10. Add further KB 2 until clear
11. Cool
12. Add evaporated water
13. Adjust pH

Physical Data

pH (10%)	: 5.5 ± 0.1	Density @ 20°C	: 1.0 ± 0.1 g cm <sup>-3</sup>
Solids (%)	: ~ 53% (typical)	Appearance	: Clear or Hazy Gel
Odour	: Characteristic	Set Point (typical)	: 30°C
Viscosity @ 20°C	: N/A		

The product was examined by x-ray diffraction and exhibited peaks at 13.145nm (intense and sharp), 7.943nm (ill defined) and 6.355nm (small), indicating cubic symmetry, and formed a clear microemulsion on dilution or heating. The latter gave good even distribution of oil applied to skin.

**Example 2**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	15	15
"EMPICOL"® CDL30J/35 (22%)	8	35.4
"EMPIGEN"® BB (30%)	8	26.7
"EMPICOL"® 07S5 (40%)	2	5
"EMPILAN"® KB2 (100%)	6	6
"EMPILAN"® KB6 (100%)	6	6
CITRIC ACID (100%)	0.5	0.5
PERFUME (100%)	0.2	0.2
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.2	0.2
"KATHON"®	---	0.2
WATER	---	Balance
<b>TOTAL</b>	<b>45.8</b>	<b>100</b>

**Physical Data**

Appearance	: Clear Liquid/Gel	Odour	: Characteristic Odour
Solids	: 36.5% (typical)	pH (100%)	: 5.5 - 6.5 (typical)
Odour	: Characteristic	Set Point	: 20 ± 5°C
Viscosity (Carimed Rheometer @ 20°C): N/A			

The product had small angle x-ray diffraction peaks characteristic of cubic symmetry and formed a clear microemulsion on dilution with water or warming. The latter gave good even deposition of oil on skin.

**Examples 3 and 4**

The following ingredients were mixed at 60°C and cooled to form ringing gels:

Component	1		2	
	Solids (%)	w/w (%)	Solids (%)	w/w (%)
"EMPIGEN"® CDL50J/35 (22%)	8	36.4	8	36.4
"EMPIGEN"® BB (30%)	8	26.7	8	26.7
"EMPICOL"® LB40 (40%)	4	7.5	3	7.5
"EMPICOL"® CVH (90%)	4	4	---	---
"EMPILAN"® KB2 (100%)	5.5	5.5	6	6
TRIETHANOLAMINE (100%)	1.1	1.1	---	---
CITRIC ACID	1	0.75	0.75	0.75
ETHYLENE DIAMINE				
TETRACETIC ACID	0.05	0.05	0.05	0.05
"KATHON"® CG (100%)	0.05	0.05	0.05	0.05
LIGHT MINERAL (100%)	14	14	20	20
WATER	---	Balance	---	Balance
<b>TOTAL</b>	<b>45.7</b>	<b>100</b>	<b>46.1</b>	<b>100</b>
<b>Appearance</b>	Clear Gel		Clear Gel	

The products in each case exhibited cubic symmetry and formed clear microemulsions or dilution with water or heating. The registered trade marks noted above have the following significance:-

- "EMPICOL" CVH is a  $C_8$  alkyl ether carboxylic acid
- "EMPICOL" LB40 is a  $C_8$   $C_{10}$  alkyl sulphate
- "EMPICOL" 0251/70J is a  $C_{12-14}$  alkyl 3 mole ethoxy sulphate
- "EMPICOL" 9758 is a  $C_{10}$  alkyl sulphate
- "EMPIGEN" BB is a  $C_{12-14}$  alkyl betaine
- "EMPIGEN" CDL is coconut amino acetate
- "EMPILAN" KB2 is a  $C_{12-14}$  alkyl 2 mole ethoxylate
- "EMPILAN" KB6 is a  $C_{12-14}$  alkyl 6 mole ethoxylate
- "GLUCAPON" 215CS is a  $C_{4-10}$  alkyl polyclucoside D.P. 1.5
- "KATHON" CG is a proprietary biocide

# Exhibit 2

# Johnson & Johnson

OFFICE OF  
GENERAL COUNSEL

ONE JOHNSON & JOHNSON PLAZA  
NEW BRUNSWICK, N.J. 08933-7002

8 June 2000

**URGENT VIA FACSIMILE 9-011-121-420-5437**

Mr. Roger Savidge  
Albright & Wilson Surfactants, Europe  
201-222 Hagley Road  
Oldbury West Midlands B68 ONN  
England

Re: PCT Patent Application Based upon UK provisional  
Patent Application No. 9913408.2 filed 10 June 1999  
For Ringing Gel Technology

Dear Roger:

As we discussed this morning, please file a Request for the above referenced application on 9 June 2000 and designate all member countries. If necessary, you may file the attached Appointment with the Request. After this application is filed, we can then select a mutually acceptable independent counsel for prosecuting this application.

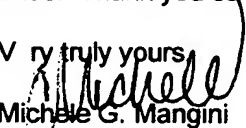
We agree that the PCT application should be filed in the names of Rhodia Consumer Specialties Limited trading as Albright & Wilson Surfactants Europe and Johnson & Johnson Consumer Companies, Inc., having an address at Grandview Road, Skillman, New Jersey 08558 USA. It is our understanding that the following individuals shall be included as inventors to this application:

1) Elvin Lukenbach  
160 Klinesville Road  
Flemington, New Jersey 08822  
USA  
United States Citizen

2) Laura McCulloch  
18 Hampton Court  
Basking Ridge, New Jersey  
USA  
United Kingdom Citizen

3) Benjamin Wiegand  
2028 Farmview Drive  
Newton, Pennsylvania 18940  
USA  
United States Citizen

In view of the fact that I will be away from the office on the 9<sup>th</sup>, please contact my assistant, Emilie Liberatore at 732 524-2820 should you need further assistance. Thank you so much for your assistance in this matter.

Very truly yours,  
  
Michele G. Mangini

**APPOINTMENT OF REPRESENTATIVE FOR INTERNATIONAL APPLICATION**

PRIORITY APPLICATION NUMBER: GB 9913408.2

PRIORITY DATE CLAIMED: 10 June 1999

TITLE OF INVENTION: PERSONAL CARE FORMULATIONS

APPLICANTS: JOHNSON & JOHNSON CONSUMER COMPANIES, INC. AND  
RHODIA CONSUMER SPECIALTIES LIMITED TRADING AS ALBRIGHT & WILSON  
SURFACTANTS EUROPE

---

The undersigned applicant hereby appoints

Mr. Roger Savidge  
of Rhodia Consumer Specialties Limited  
trading as Albright & Wilson Surfactants Europe  
201-222 Hagley Road  
Oldbury West Midlands B68 0NN  
England

To act on their behalf before the competent International Authorities in connection with  
this international application and to receive payments on their behalf.

New Brunswick, NJ USA  
New Brunswick, NJ USA

8 June 2000  
8 June 2000

Johnson & Johnson Consumer Companies, Inc.  
Johnson & Johnson Consumer Companies, Inc.

---

By: Michele Gaika Mangini  
Assistant Secretary

RECEIVED

APR 17 2002

OFFICE OF PETITIONS



# Exhibit 3

# PCT

## REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference  
(if desired) (12 characters maximum) MPD315/PCT/RGMS

**Box No. I TITLE OF INVENTION**  
PERSONAL CARE FORMULATIONS

**Box No. II APPLICANT**

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

RHODIA CONSUMER SPECIALTIES LIMITED TRADING AS ALBRIGHT  
& WILSON SURFACTANTS EUROPE AND JOHNSON & JOHNSON  
CONSUMER COMPANIES INC  
210-222 HALGEY ROAD WEST  
OLDBURY, WEST MIDLANDS, B68 0NN  
GREAT BRITAIN

☐ This person is also inventor.

Telephone No.  
+44 121 420 5430

Facsimile No.  
+44 121 420 5437

Teleprinter No.  
336291 ALBRIW G

State (that is, country) of nationality:  
GB

State (that is, country) of residence:  
GB

This person is applicant  
for the purposes of:

☐ all designated  
States

☐ all designated States except  
the United States of America

☐ the United States  
of America only

☐ the States indicated in  
the Supplemental Box

**Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)**

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

HATCHMAN Kevan  
5 Byland Close  
Friarscroft  
Bromsgrove  
Worcestershire, B61 7PL GREAT BRITAIN

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (If this check-box  
is marked, do not fill in below.)

State (that is, country) of nationality:  
GB

State (that is, country) of residence:  
GB

This person is applicant  
for the purposes of:

☐ all designated  
States

☐ all designated States except  
the United States of America

☒ the United States  
of America only

☐ the States indicated in  
the Supplemental Box

☐ Further applicants and or (further) inventors are indicated on a continuation sheet.

**Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE**

The person identified below is hereby has been appointed to act on behalf  
of the applicant(s) before the competent International Authorities as:

☒ agent

☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

SAVIDGE Roger Gordon Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury, West Midlands  
B68 0NN  
GREAT BRITAIN

Telephone No.  
+44 121 420 5430

Facsimile No.  
+44 121 420 5437

Teleprinter No.  
336291 ALBRIW G

☐ Address for correspondence: Mark this check-box where no agent or common representative is has been appointed and the  
space above is used instead to indicate a special address to which correspondence should be sent.

## Continuation of Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

If none of the following sub-boxes is used, this sheet should not be included in the request.

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

LUKENBACH Elvin  
160 Kinesville Road  
Flemington, New Jersey 08822  
UNITED STATES OF AMERICA

This person is:

- ☐ applicant only  
☒ applicant and inventor  
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

US

State (that is, country) of residence:

US

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☒ the United States of America only☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

MCCULLOCH Laura  
18 Hampton Court  
Basking Ridge, New Jersey  
UNITED STATES OF AMERICA

This person is:

- ☐ applicant only  
☒ applicant and inventor  
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

GB

State (that is, country) of residence:

US

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☒ the United States of America only☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

WIEGAND Benjamin  
2028 Farmview Drive  
Newton, Pennsylvania 18940  
UNITED STATES OF AMERICA

This person is:

- ☐ applicant only  
☒ applicant and inventor  
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

US

State (that is, country) of residence:

US

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☒ the United States of America only☐ the States indicated in the Supplemental Box

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

This person is:

- ☐ applicant only  
☐ applicant and inventor  
☐ inventor only (If this check-box is marked, do not fill in below.)

State (that is, country) of nationality:

State (that is, country) of residence:

This person is applicant for the purposes of:

☐ all designated States☐ all designated States except the United States of America☐ the United States of America only☐ the States indicated in the Supplemental Box☐ Further applicants and/or (further) inventors are indicated on another continuation sheet.

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes: at least one must be marked):

## Regional Patent

- ☐ AP ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SL Sierra Leone, SZ Swaziland, TZ United Republic of Tanzania, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ EA Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ EP European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ OA OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment desired, specify on dotted line)

## National Patent (if other kind of protection or treatment desired, specify on dotted line):

- |  |  |
|--|--|
| <input checked="" type="checkbox"/> AE United Arab Emirates                  | <input checked="" type="checkbox"/> LR Liberia                                   |
| <input checked="" type="checkbox"/> AL Albania                               | <input checked="" type="checkbox"/> LS Lesotho                                   |
| <input checked="" type="checkbox"/> AM Armenia                               | <input checked="" type="checkbox"/> LT Lithuania                                 |
| <input checked="" type="checkbox"/> AT Austria                               | <input checked="" type="checkbox"/> LU Luxembourg                                |
| <input checked="" type="checkbox"/> AU Australia                             | <input checked="" type="checkbox"/> LV Latvia                                    |
| <input checked="" type="checkbox"/> AZ Azerbaijan                            | <input checked="" type="checkbox"/> MA Morocco                                   |
| <input checked="" type="checkbox"/> BA Bosnia and Herzegovina                | <input checked="" type="checkbox"/> MD Republic of Moldova                       |
| <input checked="" type="checkbox"/> BB Barbados                              | <input checked="" type="checkbox"/> MG Madagascar                                |
| <input checked="" type="checkbox"/> BG Bulgaria                              | <input checked="" type="checkbox"/> MK The former Yugoslav Republic of Macedonia |
| <input checked="" type="checkbox"/> BR Brazil                                | <input checked="" type="checkbox"/> MN Mongolia                                  |
| <input checked="" type="checkbox"/> BY Belarus                               | <input checked="" type="checkbox"/> MW Malawi                                    |
| <input checked="" type="checkbox"/> CA Canada                                | <input checked="" type="checkbox"/> MX Mexico                                    |
| <input checked="" type="checkbox"/> CH and LI Switzerland and Liechtenstein  | <input checked="" type="checkbox"/> NO Norway                                    |
| <input checked="" type="checkbox"/> CN China                                 | <input checked="" type="checkbox"/> NZ New Zealand                               |
| <input checked="" type="checkbox"/> CR Costa Rica                            | <input checked="" type="checkbox"/> PL Poland                                    |
| <input checked="" type="checkbox"/> CU Cuba                                  | <input checked="" type="checkbox"/> PT Portugal                                  |
| <input checked="" type="checkbox"/> CZ Czech Republic                        | <input checked="" type="checkbox"/> RO Romania                                   |
| <input checked="" type="checkbox"/> DE Germany                               | <input checked="" type="checkbox"/> RU Russian Federation                        |
| <input checked="" type="checkbox"/> DK Denmark                               | <input checked="" type="checkbox"/> SD Sudan                                     |
| <input checked="" type="checkbox"/> DM Dominica                              | <input checked="" type="checkbox"/> SE Sweden                                    |
| <input checked="" type="checkbox"/> EE Estonia                               | <input checked="" type="checkbox"/> SG Singapore                                 |
| <input checked="" type="checkbox"/> ES Spain                                 | <input checked="" type="checkbox"/> SI Slovenia                                  |
| <input checked="" type="checkbox"/> FI Finland                               | <input checked="" type="checkbox"/> SK Slovakia                                  |
| <input checked="" type="checkbox"/> GB United Kingdom                        | <input checked="" type="checkbox"/> SL Sierra Leone                              |
| <input checked="" type="checkbox"/> GD Grenada                               | <input checked="" type="checkbox"/> TJ Tajikistan                                |
| <input checked="" type="checkbox"/> GE Georgia                               | <input checked="" type="checkbox"/> TM Turkmenistan                              |
| <input checked="" type="checkbox"/> GH Ghana                                 | <input checked="" type="checkbox"/> TR Turkey                                    |
| <input checked="" type="checkbox"/> GM Gambia                                | <input checked="" type="checkbox"/> TT Trinidad and Tobago                       |
| <input checked="" type="checkbox"/> HR Croatia                               | <input checked="" type="checkbox"/> TZ United Republic of Tanzania               |
| <input checked="" type="checkbox"/> HU Hungary                               | <input checked="" type="checkbox"/> UA Ukraine                                   |
| <input checked="" type="checkbox"/> ID Indonesia                             | <input checked="" type="checkbox"/> UG Uganda                                    |
| <input checked="" type="checkbox"/> IL Israel                                | <input checked="" type="checkbox"/> US United States of America                  |
| <input checked="" type="checkbox"/> IN India                                 | <input checked="" type="checkbox"/> UZ Uzbekistan                                |
| <input checked="" type="checkbox"/> IS Iceland                               | <input checked="" type="checkbox"/> VN Viet Nam                                  |
| <input checked="" type="checkbox"/> JP Japan                                 | <input checked="" type="checkbox"/> YU Yugoslavia                                |
| <input checked="" type="checkbox"/> KE Kenya                                 | <input checked="" type="checkbox"/> ZA South Africa                              |
| <input checked="" type="checkbox"/> KG Kyrgyzstan                            | <input checked="" type="checkbox"/> ZW Zimbabwe                                  |
| <input checked="" type="checkbox"/> KP Democratic People's Republic of Korea |  |
| <input checked="" type="checkbox"/> KR Republic of Korea                     |  |
| <input checked="" type="checkbox"/> KZ Kazakhstan                            |  |
| <input checked="" type="checkbox"/> LC Saint Lucia                           |  |
| <input checked="" type="checkbox"/> LK Sri Lanka                             |  |

Check-boxes reserved for designating States which have become party to the PCT after issuance of this sheet:

- ☒ .AG .ANTIGUA. & .BARBUDA
- ☒ .DZ .ALGERIA

Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation (including fees) must reach the receiving Office within the 15-month time limit.)

**Supplemental Box**

If the Supplemental Box is not used, this sheet should not be included in the request.

If, in any of the Boxes, the space is insufficient to furnish all the information: in such case, write "Continuation of Box No. ..." (indicate the number of the Box) and furnish the information in the same manner as required according to the captions of the Box in which the space was insufficient, in particular:

- (i) if more than two persons are involved as applicants and/or inventors and no "continuation sheet" is available: in such case, write "Continuation of Box No. III" and indicate for each additional person the same type of information as required in Box No. III. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below;
- (ii) if, in Box No. II or in any of the sub-boxes of Box No. III, the indication "the States indicated in the Supplemental Box" is checked: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the applicant(s) involved and, next to (each) such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is applicant;
- (iii) if, in Box No. II or in any of the sub-boxes of Box No. III, the inventor or the inventor/applicant is not inventor for the purposes of all designated States or for the purposes of the United States of America: in such case, write "Continuation of Box No. II" or "Continuation of Box No. III" or "Continuation of Boxes No. II and No. III" (as the case may be), indicate the name of the inventor(s) and, next to (each) such name, the State(s) (and/or, where applicable, ARIPO, Eurasian, European or OAPI patent) for the purposes of which the named person is inventor;
- (iv) if, in addition to the agent(s) indicated in Box No. IV, there are further agents: in such case, write "Continuation of Box No. IV" and indicate for each further agent the same type of information as required in Box No. IV;
- (v) if, in Box No. V, the name of any State (or OAPI) is accompanied by the indication "patent of addition," or "certificate of addition," or if, in Box No. V, the name of the United States of America is accompanied by an indication "continuation" or "continuation-in-part": in such case, write "Continuation of Box No. V" and the name of each State involved (or OAPI), and after the name of each such State (or OAPI), the number of the parent title or parent application and the date of grant of the parent title or filing of the parent application;
- (vi) if, in Box No. VI, there are more than three earlier applications whose priority is claimed: in such case, write "Continuation of Box No. VI" and indicate for each additional earlier application the same type of information as required in Box No. VI;
- (vii) if, in Box No. VI, the earlier application is an ARIPO application: in such case, write "Continuation of Box No. VI", specify the number of the item corresponding to that earlier application and indicate at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed.

2. If, with regard to the precautionary designation statement contained in Box No. V, the applicant wishes to exclude any State(s) from the scope of that statement: in such case, write "Designation(s) excluded from precautionary designation statement" and indicate the name or two-letter code of each State so excluded.

3. If the applicant claims, in respect of any designated Office, the benefits of provisions of the national law concerning non-prejudicial disclosures or exceptions to lack of novelty: in such case, write "Statement concerning non-prejudicial disclosures or exceptions to lack of novelty" and furnish that statement below.

**CONTINUATION OF BOX NO. V****Designations (continued)**

x MZ MOZAMBIQUE

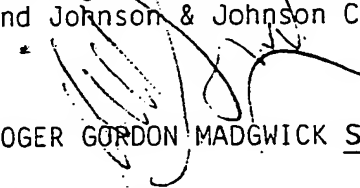
<b>Box No. VI PRIORITY CLAIM</b>		<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.		
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application: regional Office	international application: receiving Office
item (1) 10 JUNE 1999	9913308.2	GB		
item (2)				
item (3)				

☐ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s):

\* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

<b>Box No. VII INTERNATIONAL SEARCHING AUTHORITY</b>		
<b>Choice of International Searching Authority (ISA)</b> (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):	<b>Request to use results of earlier search: reference to that search</b> (if an earlier search has been carried out by or requested from the International Searching Authority): Date (day/month/year)      Number      Country (or regional Office)	
ISA / EP		

<b>Box No. VIII CHECK LIST: LANGUAGE OF FILING</b>	
This international application contains the following number of sheets: request : 5 description (excluding sequence listing part) : 25 claims : 1 abstract : 1 drawings : sequence listing part of description : Total number of sheets : 32	This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input type="checkbox"/> separate signed power of attorney TO FOLLOW 3. <input checked="" type="checkbox"/> copy of general power of attorney; reference number, if any: & copy of General Power of Signatory. 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): TO FOLLOW 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input checked="" type="checkbox"/> other (specify): Change of Name Certificate
Figure of the drawings which should accompany the abstract: N/A	Language of filing of the international application: ENGLISH

<b>Box No. IX SIGNATURE OF APPLICANT OR AGENT</b>	
Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request). for and on behalf of Rhodia Consumer Specialties Limited trading as Albright & Wilson Surfactants Europe and Johnson & Johnson Consumer Companies Inc   ROGER GORDON MADGWICK SAVIDGE - By Power of Attorney	

For receiving Office use only	
1. Date of actual receipt of the purported international application: 3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application: 4. Date of timely receipt of the required corrections under PCT Article 11(2): 5. International Searching Authority (if two or more are competent): ISA /	2. Drawings: <input type="checkbox"/> received: <input type="checkbox"/> not received: 6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.

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Date of receipt of the record copy by the International Bureau:

# PCT

## FEE CALCULATION SHEET Annex to the Request

For receiving Office use only

International application No. \_\_\_\_\_

Applicant's or agent's  
file reference

MPD315/PCT/RGMS

Date stamp of the receiving Office

Applicant

RHODIA CONSUMER SPECIALTIES LIMITED TRADING AS ALBRIGHT & WILSON  
SURFACTANTS EUROPE AND JOHNSON & JOHNSON CONSUMER COMPANIES INC

### CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE

DEM 199.49

T

2. SEARCH FEE

DEM 1848.26

S

International search to be carried out by \_\_\_\_\_

(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FEE

#### Basic Fee

The international application contains 32 sheets.

first 30 sheets

DEM 806.51

b1

2 remaining sheets

x 19.95 additional amount

DEM 39.90

b2

Add amounts entered at b1 and b2 and enter total at B

DEM 846.41

B

#### Designation Fees

ALL STATES DESIGNATED

The international application contains \_\_\_\_\_ designations.

8 number of designation fees payable (maximum 10)

x DEM 148.64 amount of designation fee

DEM 1189.12

D

Add amounts entered at B and D and enter total at I

DEM 2035.53

I

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicant is (or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT (if applicable)

P

5. TOTAL FEES PAYABLE

DEM 4083.28

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

TOTAL

☐ The designation fees are not paid at this time.

### MODE OF PAYMENT

☒ authorization to charge deposit account (see below)

☐ bank draft

☐ coupons

☐ cheque

☐ cash

☐ other (specify):

☐ postal money order

☐ revenue stamps

### DEPOSIT ACCOUNT AUTHORIZATION (this mode of payment may not be available at all receiving Offices)

The RO EP ☒ is hereby authorized to charge the total fees indicated above to my deposit account.

☒ (this check-box may be marked only if the conditions for deposit accounts of the receiving Office so permit) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

☐ is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

28 05 00 09

9 JUNE 2000

(GA235)

Deposit Account No.

Date (day month year)

Signature Roger Gordon Madgwick SAVIDGE

# **APPOINTMENT OF REPRESENTATIVE FOR INTERNATIONAL APPLICATION**

PRIORITY APPLICATION NUMBER: GB 9913408.2

PRIORTY DATE CLAIMED: 10 June 1999

TITLE OF INVENTION: PERSONAL CARE FORMULATIONS

APPLICANTS: JOHNSON & JOHNSON CONSUMER COMPANIES, INC. AND  
RHODIA CONSUMER SPECIALTIES LIMITED TRADING AS ALBRIGHT & WILSON  
SURFACTANTS EUROPE

The undersigned applicant hereby appoints

Mr. Roger Savidge  
of Rhodia Consumer Specialties Limited  
trading as Albright & Wilson Surfactants Europe  
201-222 Hagley Road  
Oldbury West Midlands B68 ONN  
England

To act on their behalf before the competent International Authorities in connection with  
this international application and to receive payments on their behalf.

New Brunswick, NJ USA  
New Brunswick, NJ USA

8 June 2000  
8 June 2000

Johnson & Johnson Consumer Companies, Inc.  
By: Michele Galka Mangini  
Assistant Secretary



**BY THIS POWER OF ATTORNEY** given this 20 day of April two thousand, **RHODIA CONSUMER SPECIALTIES LIMITED**, a Company duly incorporated in England and having its Registered Office at 21Q-222 Hagley Road West, Oldbury, West Midlands (hereinafter called "the Company") hereby appoints **ROGER GORDON MADGWICK SAVIDGE** (hereinafter called "the Attorney") to be the true and lawful Agent and Attorney of the Company on behalf of and in the name of the Company or otherwise to do, perform, exercise and execute or concur with any other person or persons in doing, performing, exercising and executing in any country or countries or jurisdiction in any part of the world all or any other the following powers, acts, deeds and things, that is to say:-

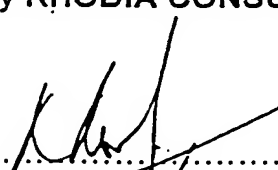
1. To make application or to cause application to be made for the grant to the Company of any letters patents, trade mark, trade name or design and the proper registration thereof and to take all steps necessary for the same to be prosecuted and maintained;
2. As the act and deed of the Company to sign, seal, deliver and execute all or any assignments or assurances to the Company of any letters patent, registered trade mark, trade name or registered design or any application therefore for the purpose of fully and effectually vesting and transferring the same into the name of the Company;
3. As the act and deed of the Company to sign, seal, deliver and execute all or any assignments, assurances, licences or sub-licences from the Company of or under any letters patent, registered trade mark, trade name or registered design or any application therefor for the purpose of fully and effectually vesting, transferring or granting the same into the name of a company (whether in the United Kingdom or elsewhere) which is a subsidiary or holding company of the Company insofar as such documents can be executed without the Company's Seal being affixed thereto;

4. To sign and execute all documents relating to applications for letters patent, registered trade marks, trade names or registered design or the renewal thereof or to assignments or assurances of letters patent or applications therefor;
5. To act in regard to all official communications which may now or hereafter be addressed to the Attorney relating to applications for letters patents, registered trade marks, trade names or registered designs or the renewal thereof in such manner that the Attorney may be recognised as the authorised Agent of the Company in all proceedings incident thereto;
6. For or in connection with any letters patent, registered trade mark, trade name or registered design or application therefor to sign, seal, deliver and execute any Power or Attorney or other deed or document;
  - a) authorising any firm of patent agents or trade mark agents in the United Kingdom of Great Britain and Northern Ireland to act on behalf of the Company;
  - b) authorising any person, persons, firm or company practising as patent agents or trade mark agents or otherwise entitled to act as agents for all matters relating to trade marks or trade names outside the United Kingdom of Great Britain and Northern Ireland to act on behalf of the Company;
7. To initiate or cause to be initiated in any Patent Office or Registry or any Trade Mark Registry or other official agency or government department or otherwise responsible for the registration or protection of trade marks, trade names or designs any proceedings or application whatsoever relating to any proprietary rights whether in the name of the Company or not and to cause such proceedings or application to be maintained or withdrawn;

**AND THE COMPANY HEREBY RATIFIED AND CONFIRMS** and agrees to ratify and confirm all and whatsoever the Attorney shall lawfully do or have done by virtue of the authorities herein contained.

**IN WITNESS WHEREOF** the Company has executed this document as a deed the day and year first above written.

Executed by **RHODIA CONSUMER SPECIALTIES LIMITED** as a deed and signed by:

Director ..... 

Secretary ..... 

# PCT

## GENERAL POWER OF ATTORNEY

(for several international applications filed under the Patent Cooperation Treaty)

(PCT Rule 90.5)

The undersigned person(s):

(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

ALBRIGHT & WILSON UK LIMITED  
P O BOX 3  
210-222 HAGLEY ROAD WEST  
OLDBURY  
WARLEY  
WEST MIDLANDS B68 0NN  
ENGLAND

hereby appoint(s) the following person(s):

☒ agents

☐ common representative

Name and address

(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

1. SAVIDGE, Roger Gordon Madgwick
2. KINTON, Colin David

both of

ALBRIGHT & WILSON UK LIMITED  
PATENTS DEPARTMENT  
P O BOX 3

210-222 HAGLEY ROAD WEST, OLDBURY, WARLEY, WEST MIDLANDS B68 0NN, ENGLAND

to represent the undersigned before

☒ all the competent International Authorities

☐ the International Searching Authority only

☐ the International Preliminary Examining Authority only

in connection with any and all international applications filed by the undersigned with the following Office

EUROPEAN PATENT OFFICE

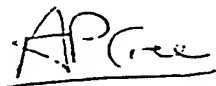
as receiving Office

and to make or receive payments on behalf of the undersigned.

Signature(s) (where there are several persons, each of them must sign; next to each signature, indicate the name of the person signing and the capacity in which the person signs, if such capacity is not obvious from reading this power):

for and on behalf of

ALBRIGHT & WILSON UK LIMITED



A P CREE, Company Secretary

Date:

27 February 1995



**CERTIFICATE OF INCORPORATION  
ON CHANGE OF NAME**

Company No. 36833

The Registrar of Companies for England and Wales hereby certifies that

ALBRIGHT & WILSON UK LIMITED

having by special resolution changed its name, is now incorporated  
under the name of

RHODIA CONSUMER SPECIALTIES LIMITED

Given at Companies House, London, the 10th March 2000

MR J MAYNE

For The Registrar Of Companies



C O M P A N I E S H O U S E

Sent to EPO  
9/1/2009

MPD315/PCT

**PATENT COOPERATIONA TREATY**

**FINAL SPECIFICATION**  
**(Description, Abstract and Claims)**

**Applicant :**

**RHODIA CONSUMER SPECIALTIES LIMITED**  
**TRADING AS ALBRIGHT & WILSON**  
**SURFACTANTS EUROPE**

**-AND-**

**JOHNSON & JOHNSON**  
**CONSUMER COMPANIES INC**

**Inventprs :**

**KEVAN HATCHMAN**  
**ELVIN LUKENBACH**  
**LAURA MCCULLOCH**  
**BENJAMIN WIEGAND**

ABSTRACT

Personal care compositions contain at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (a) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (b) a hydrophilic surfactant having an HLB greater than 11 in a weight proportion of from 1:1 to 1:30 based on the weight of (a), said water surfactant and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.

### PERSONAL CARE FORMULATIONS

The present invention relates to shampoo or cleaning compositions suitable for personal care applications in the form of  $I_1$  mesophase systems containing dispersed oil.

Dispersing oil in aqueous shampoo and body wash formulations has presented problems. To prevent the oil phase separating it must either be: (A) emulsified which involves dispersing the oil as colloidal single droplets; (B) microemulsified which involves forming a micellar solution with oil incorporated into surfactant micelles; (C) suspended in a structured surfactant system which typically comprises a dispersion of a surfactant mesophase in aqueous electrolyte; or (D) incorporated into a water soluble solid, pasty or gelatinous composition.

With the exception of microemulsions which are clear, thermodynamically stable, micellar solutions, the foregoing systems are necessarily opaque and contain the oil dispersed in a relatively coarse form, which does not deposit satisfactorily on skin or hair.

However microemulsions are difficult to formulate using the surfactants which are most effective in body wash and other personal care formulations and contain relatively low concentrations of surfactant.

We have now discovered that oil may be stably incorporated into the structure of an  $I_1$  phase to form a clear gel-like composition which contains higher concentrations of surfactant and oil than conventional microemulsions, but which dissolves in water to form a microemulsion. The novel oil-in- $I_1$  compositions also form microemulsions on heating.



Surfactants are known to form mesophases or liquid crystal phases at concentrations above approximately 30% by weight based on the weight of water and surfactant. Mesophases are phases which exhibit a degree of order intermediate between typical liquids and solids. Generally mesophases combine long range order associated with crystals, with fast molecular motion common to liquids.

The formation of detergent mesophases is well documented. Different surfactants and surfactant mixtures differ widely in their ability to form the numerous different mesophases, and in respect of the conditions of concentration and temperature at which they are formed. For a typical surfactant of the type normally used in cleaning products the following mesophases are usually observed. The concentrations given are illustrative only and may vary considerably from one surfactant or surfactant mixture to the next.

Below approximately 30% surfactant an isotropic  $L_1$  phase is formed (with micelles of surfactant in water). Above 30% surfactant many detergents form a M phase which is not normally used in personal care applications since it does not show suitable flow characteristics and is difficult to dissolve or disperse in water. Above the concentrations required to form an M phase, but usually at concentrations of less than 80% active surfactant, i.e. 60%-80% a G-phase is formed. At concentrations higher than those required to form a G-phase, i.e. typically greater than 80% active surfactant, most surfactants form a hydrated solid, and some, especially non-ionic surfactants form a liquid phase containing dispersed micelle sized droplets of water - an inverted micellar solution known as an  $L_2$  phase.  $L_2$  detergent systems do not disperse readily in water and have a tendency to form undesirable gels, e.g. M phases, on dilution.

Some surfactants form viscous isotropic or VI phases. These are immobile phases usually with a vitreous appearance, and have been relatively little studied compared to the other phases discussed above. They have been virtually ignored in the context of formulating cleaning compositions because most of the surfactants and surfactant systems which are commonly used in cleaning compositions do not form VI phases, at least at

normal temperatures, or form them only within narrow concentration ranges and because their known properties as immobile gels has deterred formulators from investigating them. They are recognised as being the most viscous of the lyotropic mesophases.

The different surfactant phases can be recognised by a combination of appearance, rheology, textures under the microscope, electron microscopy and x-ray diffraction or neutron scattering. A detailed description, with illustrations, of the difference textures observable using a polarising microscope, is to be found in the paper by Rosevear JAOCS Vol 31, p628.

The following terms may require explanation or definition:

The "hydrophilic: lipophilic balance", or "HLB" value is used as a measure of the relative affinities of the surfactants for water and oil respectively and correlates with their effectiveness as emulsifiers. HLB value can easily be calculated for alcohol ethoxylates since it is one fifth of the weight percent of ethylene oxide based on the total mole weight. Other surfactants can be assigned equivalent values by applying more complicated formulae or by measuring their relative affinity for water and oil. An HLB value of 20 represents a completely water soluble oil insoluble surfactant, while an HLB value of 0 represents a completely oil soluble and water insoluble surfactant.

"Optically isotropic" surfactant phases do not normally tend to rotate the plane of polarisation of plane polarised light. If a drop of sample is placed between two sheets of optically plane polarising material whose planes are at right angles, and light is shone on to one sheet, optically isotropic surfactant samples do not appear substantially brighter than their surrounding when viewed through the other sheet. Optically anisotropic materials appear substantially brighter. Optically anisotropic mesophases typically show characteristic textures when viewed through a microscope between crossed polarisers, whereas optically isotropic phases usually show a featureless continuum.

"Newtonian liquids" have a viscosity which remains constant at different shear rates. For the purpose of this specification, liquids are considered Newtonian if the viscosity does not vary substantially at shear rates up to  $1000 \text{ sec}^{-1}$ .

"Lamellar" phases are phases which comprise a plurality of bilayers of surfactant arranged in parallel and separated by liquid medium. They include both solid phases and the typical form of the liquid crystal G-phase. G-phases are typically pourable, non-Newtonian, anisotropic products. They are typically viscous-looking, opalescent materials with a characteristic "smeary" appearance on flowing. They form characteristic texture under the polarising microscope and freeze fractured samples have a lamellar appearance under the electron microscope. X-ray diffraction or neutron scattering similarly reveal a lamellar structure, with a principal peak typically between 4 and 10nm, usually 5 to 6nm. Higher order peaks, when present occur at double or higher integral multiples of the Q value of the principal peak. Q is the momentum transfer vector and is related, in the case of lamellar phases, to the repeat spacing d by the equation  $Q = \frac{2n}{d} [\pi]$  where n is the order of the peak.

G-phases, however, can exist in several different forms, including domains of parallel sheets which constitute the bulk of the typical G-phases described above and spherulites formed from a number of concentric spheroidal shells, each of which is a bilayer of surfactant. In this specification the term "lamellar" will be reserved for compositions which are at least partly of the former type. Opaque compositions at least predominantly of the latter type in which the continuous phase is a substantially isotropic solution containing dispersed spherulites are referred to herein as "G-phase compositions". G-phases are sometimes referred to in the literature as  $L_{(\alpha)}$  phases.

$L_1$ -phases are mobile, optically isotropic, and typically Newtonian liquids which show no texture under the polarising microscope. Electron microscopy is capable of resolving the texture of such phases only at very high magnifications, and X-ray or neutron scattering normally gives only a single broad peak typical of a liquid structure, at very small angles

close to the reference beam. The viscosity of an  $L_1$ -phase is usually low, but may rise significantly as the concentration approaches the upper phase boundary.

"M-phases" are typically immobile, anisotropic products resembling low melting point waxes. They give characteristic textures under the polarising microscope, and a hexagonal diffraction pattern by X-ray or neutron diffraction which comprises a major peak, usually at values corresponding to a repeat spacing between 4 and 10nm, and sometimes higher order peaks, the first at a Q-value which is  $3^{0.5}$  times the Q-value of the principal peak and the next double the Q-value of the principal peak. M-phases are sometimes referred to in the literature as H-phases.

The viscous isotropic or "VI" phases are typically immobile, non-Newtonian, optically isotropic and are typically transparent, at least when pure. VI phases have a cubic symmetrical diffraction pattern, under X-ray diffraction or neutron scattering, with a principal peak and higher order peaks at  $2^{0.5}$  and  $3^{0.5}$  times the Q-value of the principal peak.

These cubic liquid crystalline phases are sometimes observed immediately following the micellar phase at ambient temperature as the concentration of surfactant is increased. It has been proposed that such VI phases, sometimes referred to as  $I_1$  phase, may arise from the packing of micelles (probably spherical) in a cubic lattice. At ambient temperature a further increase in surfactant concentration usually results in hexagonal phase ( $M_1$ ), which may be followed by a lamellar phase (G).  $I_1$  phases, when they occur, are usually only observed over a narrow range of concentrations, typically just above those at which the  $L_1$ -phase is formed. The location of such VI phases in a phase diagram suggests that the phase is built up of small closed surfactant aggregates in a water continuum.

An inverse form of the  $I_1$  phase (the  $I_2$  phase) has also been reported, possibly between the inverse hexagonal ( $M_2$ ) and  $L_2$  phases. It consists of a surfactant continuum containing a cubic array of inverted micelles. An alternative form of the VI phase called

the  $V_1$  phase has been observed at concentrations between the M and  $G_1$  phases and may comprise a bicontinuous system. This may exhibit an even higher viscosity than the  $I_1$ . An inverse phase, the  $V_2$  phase, between the G and  $M_2$  phases has also been postulated.

VI phases are typically examples of "ringing gels". When a jar or beaker containing such a phase is sharply struck, a distinctive vibration can be felt in the composition.

The  $I_1/L_1$  transition temperature will be referred to herein as the melting point of the  $I_1$  phase for convenience, although it is not strictly speaking the melting point since the VI phases are not solids.

All references herein to the formation or existence of specific phases or structures are to be construed, unless the context requires otherwise, as references to their formation or existence at 20°C.

Hexagonal gels (M-phase) have been referred to in the prior art as cleaning compositions, e.g. GB 2 179 055, EP I 153 837 and colloidal gels formed with gelling agents such as synthetic polymers or gelatin have also been suggested, e.g. US 4 465 663.

However these compositions cannot be readily dissolved in water to form microemulsions. They are moreover usually opaque and of an unattractive appearance and often require the presence of solvents such as glycols which add to the cost and are environmentally undesirable.

The use of a type of ringing gel to suspend oil for cosmetic or pharmaceutical applications was described in US 4 026 818 but the formulation requires the presence of hydroxylic solvents and utilises a surfactant system which is unsuitable for shampoo applications. EP O 598 335 describes the use of various cubic phases including  $I_1$  phases as laundry prespotters and for other cleaning formulations. It does not suggest how such phases could be used to suspend oil or form microemulsions. Normally attempts to

suspend oil in surfactant mesophases result in coarse droplets of oil being suspended in the aqueous phase of a structured surfactant.

Our invention provides a concentrated personal cleansing composition comprising, by weight of the composition, at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.

The surfactants are preferably selected to provide an  $I_1$  phase over a comparatively broad surfactant concentration range e.g. more than  $\pm 5\%$  or greater, which range typically lies above 15% by weight total surfactant based on the weight of the composition e.g. between 20% and 40% by weight surfactant usually between 25% and 60%.

The surfactants are preferably selected to provide an  $I_1$  phase which melts above 30°C e.g. above 35°C, most preferably above 40°C. Preferably the  $I_1$  phase melts at a temperature substantially below 100°C, e.g. below 90°C, more preferably below 80°C, most preferably below 70°C, especially below 60°C, typically below 55°C, usually below 50°C.

The surfactant mixture preferably has a mean HLB based on the molar proportions of the components between 10 and 15 e.g. 11 to 14. The surfactants preferably comprise non-ionic surfactants such as ethoxylated alcohols. It has been found that highly ethoxylated fatty alcohols, e.g. more than 10 EO groups, preferably more than 15 EO groups, especially 18 to 50 EO groups form  $I_1$  phases particularly readily.

Other non-ionic surfactants which may be present include:-

alkyl phenol ethoxylates, fatty acid ethoxylates, fatty acid monoalkylolamide ethoxylates, fatty alcohol propoxylates, fatty anime alkoxylates and fatty acid glyceryl ester ethoxylates. Other non-ionic compounds suitable for inclusion in compositions of the present invention include mixed ethylene oxide propylene oxide block copolymers, low relative molecular mass polyethylene glycols e.g. PEG600 and PEG200, ethylene glycol monoesters, amine oxides and alkyl polyglycosides, alkyl sugar esters including alkyl sucrose esters and alkyl oligosaccharide ester, alkyl capped polyvinyl alcohol and alkyl capped polyvinyl pyrrolidone.

Compositions of the invention may also comprise anionic surfactants, in addition to or instead of non-ionic surfactants. Anionic surfactant may comprise a C<sub>10-20</sub> alkyl benzene sulphonate or an alkyl ether sulphate which is preferably the product obtained by ethoxylating a natural fatty or synthetic C<sub>10-20</sub> e.g. a C<sub>12-14</sub> alcohol with from 1 to 20, preferably 2 to 10 e.g. 3 to 4 ethyleneoxy groups, optionally stripping any unreacted alcohol, reacting the ethoxylated product with a sulphating agent and neutralising the resulting alkyl ether sulphuric acid with a base. The term also includes alkyl glyceryl sulphates, and random or block copolymerised alkyl ethoxy/propoxy sulphates.

The anionic surfactant may also comprise, for example, C<sub>10-20</sub> e.g. C<sub>12-18</sub> alkyl sulphate.

The surfactant may comprise a C<sub>8-20</sub> e.g. C<sub>10-20</sub> aliphatic soap. The soap may be saturated or unsaturated, straight or branched chain.

Preferred examples include dodecanoates, myristates, stearates, oleates, linoleates, linoleates and palmitates and coconut and tallow soaps.

The surfactant may include other anionic surfactants, such as olefin sulphonates, paraffin sulphonates, taurides, isethionates, ether sulphonates, ether carboxylates, aliphatic ester sulphonates e.g. alkyl glyceryl sulphonates, sulphosuccinates or sulphosuccinamates.

The cation of any anionic surfactant is typically sodium but may alternatively be potassium, lithium, calcium, magnesium, ammonium, or an alkyl ammonium having up to 6 aliphatic carbon atoms including isopropyl ammonium, monoethanol ammonium, diethanol ammonium, and triethanol ammonium.

Ammonium and ethanol ammonium salts are generally more soluble than the sodium salts. Mixtures of the above cations may be used.

The composition may contain amphoteric surfactants such as betaines sulphobetaines, amido betaines or imidazoline betaines.

The  $I_1$  phase may be conveniently prepared by mixing the oil and oil soluble surfactant and adding sufficient water to the water soluble surfactant to maintain a lamellar phase. The oil and oil soluble surfactant may be stirred into the lamellar composition at elevated temperature, above the melting point of the desired  $I_1$  phase. The composition is then diluted with hot water until a microemulsion is formed and then cooled to solidify it into the  $I_1$  phase.

The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether having, for example, a boiling point below 120°C e.g. below 100°C especially below 80°C) or a lower molecular weight fatty ester (e.g. one having less than 25 carbon atoms) such as isopropyl esters of lauric isostearic or palmitic acids or their ethyl analogues. Other oils, including higher mol weight fatty esters, e.g. oleyl oleate, fatty glycerides, terpene oils such as limonene or silicone oils may present difficulties in providing clear compositions. Such oils can nevertheless be incorporated in clear formulations by blending with sufficient mineral oil (preferably low molecular weight mineral oil). The amount required varies according to the nature of the oil. Typically the blend contains at least 16%, based on the total weight of oil, of the mineral oil, especially 30 to 80%, typically 40 to 60%. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil.



Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolved in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds, ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide may be dispersed in the hot microemulsion prior to cooling. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may be suspended. Other active ingredients which may be suspended include insect repellants and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

Electrolytes tend to break  $I_1$  phase structure and are preferably present in concentrations below 10% based on total weight of the compositions, more preferably below 5%, e.g. 0 to 3%, most preferably 0 to 1%. Generally we prefer that electrolyte be substantially absent. Adventitious chloride or sulphate present as impurities in the surfactant can be tolerated. Small amounts of builder such as citrates, pyrophosphates, polyphosphates may optionally be included.

Water soluble solvents are generally undesirable and are not required to form stable  $I_1$  structures according to the invention. We therefore prefer that they should be substantially absent. Although small amounts of, for example, ethanol or propanol or of a water miscible polyhydric alcohol or alcohol ester may sometimes be desired for special purposes, they are preferably present in amounts less than 5% by weight, more preferably less than 3% by weight, most preferably less than 2% by weight, e.g. less than 1% by weight.

The composition may optionally contain hydrotropes such as sodium lower alkyl benzene sulphonate e.g. sodium toluene, xylene or cumene sulphonate or urea, however these are not generally necessary and are not generally preferred. We prefer that these should be present in quantities less than 5% by weight, more preferably less than 4%, especially less than 2% e.g. 0 to 1%. They may be useful occasionally to avoid haziness of the gel.

The total amount of water is preferably from 25 to 60% by weight of the composition, more preferably 30 to 50%, e.g. 35 to 50%. The total weight percentage of surfactant based on the weight of the composition is preferably from 15 to 35%, e.g. 20 to 30%. The proportion of oil is preferably greater than 5%, more preferably greater than 8%, e.g. 10 to 30%, especially 15 to 25% by weight based on the weight of the composition. The oil soluble surfactant is preferably present in a proportion of more than 1:5 based on the weight of oil, more preferably from 1:2 to 5:1. The oil soluble surfactant preferably has an HLB of from 3 to 9 e.g. 4 to 8.

The weight ratio of water soluble surfactant to oil soluble surfactant is preferably 1:1 to 30:1, more preferably 2:1 to 20:1, typically 3:1 to 15:1, e.g. 4:1 to 10:1. The water soluble surfactant preferably has an HLB greater than 12, more preferably greater than 13, especially 14 to 19.

The product may be cast into shaped bodies or formed into particles or granules, e.g. by spray cooling a hot solution of the  $L_1$  phase formed on melting the composition.

The composition may be converted into a microemulsion phase by addition of water, by heating above the melting point or by adding electrolyte such as salt and the invention includes  $L_1$  phases when so prepared.

The invention will be illustrated by the following examples:

**Example 1**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>w/w (%)</u></b>
MINERAL OIL (100%)	20	20
"EMPICOL"® 0251/70J (70%)	11.2	16
"EMPIGEN"® BB (30%)	4.8	16
"GLUCAPON"® 215 CS UP (65%)	6	9.2
"EMPILAN"® KB2 (100%)	7.5	7.5
SODIUM CHLORIDE (100%)	2	2
PERFUME (100%)	0.5	0.5
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.1	0.1
CITRIC ACID (100%)	0.2	0.2
BENZOIC ACID (100%)	0.3	0.3
SODIUM HYDROXIDE (47%)	0.1	0.2
WATER	---	Balance

The method of mixing comprised the following steps:-

1. Charge 50% of water
2. Heat to 60°C
3. Add EDTA, sodium benzoate, citric acid and 47% NaOH dissolve with stirring
4. Add "EMPIGEN" BB
5. Add mineral oil and disperse with stirring
6. Add "EMPILAN" KB 2 and mix thoroughly
7. Add "EMPICOL" 0251/70j
8. Add remaining water
9. Add "GLUCAPON" 215 CS UP
10. Add further KB 2 until clear
11. Cool
12. Add evaporated water
13. Adjust pH

**Physical Data**

pH (10%)	: 5.5 ± 0.1	Density @ 20°C	: 1.0 ± 0.1 g cm <sup>-3</sup>
Solids (%)	: ~ 53% (typical)	Appearance	: Clear or Hazy Gel
Odour	: Characteristic	Set Point (typical)	: 30°C
Viscosity @ 20°C : N/A			

The product was examined by x-ray diffraction and exhibited peaks at 13.145nm (intense and sharp), 7.943nm (ill defined) and 6.355nm (small), indicating cubic symmetry, and formed a clear microemulsion on dilution or heating. The latter gave good even distribution of oil applied to skin.

**Example 2**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	15	15
"EMPICOL"® CDL30J/35 (22%)	8	35.4
"EMPIGEN"® BB (30%)	8	26.7
"EMPICOL"® 0785 (40%)	2	5
"EMPILAN"® KB2 (100%)	6	6
"EMPILAN"® KB6 (100%)	6	6
CITRIC ACID (100%)	0.5	0.5
PERFUME (100%)	0.2	0.2
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.2	0.2
"KATHON"®	---	0.2
WATER	---	Balance
<b>TOTAL</b>	<b>45.8</b>	<b>100</b>

#### Physical Data

Appearance	: Clear Liquid/Gel	Odour	: Characteristic Odour
Solids	: 36.5% (typical)	pH (100%)	: 5.5 - 6.5 (typical)
Odour	: Characteristic	Set Point	: 20 ± 5°C

Viscosity (Carfimed Rheometer @ 20°C : N/A

The product had small angle x-ray diffraction peaks characteristic of cubic symmetry and formed a clear microemulsion on dilution with water or warming. The latter gave good even deposition of oil on skin.

**Examples 3 and 4**

The following ingredients were mixed at 60°C and cooled to form ringing gels:

Component	1		2	
	Solids (%)	w/w (%)	Solids (%)	w/w (%)
"EMPIGEN"® CDL30J/35 (22%)	8	36.4	8	36.4
"EMPIGEN"® BB (30%)	8	26.7	8	26.7
"EMPICOL"® LB40 (40%)	4	7.5	3	7.5
"EMPICOL"® CVH (90%)	4	4	---	---
"EMPILAN"® KB2 (100%)	5.5	5.5	6	6
TRIETHANOLAMINE (100%)	1.1	1.1	---	---
CITRIC ACID	1	0.75	0.75	0.75
ETHYLENE DIAMINE				
TETRACETIC ACID	0.05	0.05	0.05	0.05
"KATHON"® CG (100%)	0.05	0.05	0.05	0.05
LIGHT MINERAL (100%)	14	14	20	20
WATER	---	Balance	---	Balance
<b>TOTAL</b>	<b>45.7</b>	<b>100</b>	<b>46.1</b>	<b>100</b>
<b>Appearance</b>	Clear Gel		Clear Gel	

The following ingredients were mixed at 60 °C and cooled to form a clear 'ringing' gel.

**Example 5**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	18	18
"EMPICOL" ® 0251 70 J (70 %)	12	17.2
"EMPICOL" ® CED5 FL (100 %)	5	5
"EMPILAN" ® KBE2 (100 %)	3	3
"EMPILAN" ® KB6 (100 %)	3	3
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

**Example 6**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
HEAVY MINERAL OIL ("KRISTOL" ® M70) (100 %)	18	18
"EMPICOL" ® 0251 70 J (70 %)	10.5	15
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KB2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.5	1.0
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

**Physical Data**

Density @ 20°C	: 1.0 +/- 0.1	pH (10 %)	: 5.5 +/- 0.5
Appearance:	Clear or hazy gel	Odour	: Characteristic
Set point (typical):	35 +/- 5°C	Viscosity @ 20°C:	N/A

**Method for examples 5 and 6**

- i) Charge water and heat to 60°C.
- ii) Add EDTA, sodium benzoate, citric acid and NaOH. Dissolve with stirring.
- iii) Add "EMPICOL" CED5 FL and mix thoroughly.
- iv) Add glycerol.
- v) Add NaCl and disperse with stirring.
- vi) Add "EMPILAN" KBE2 and "EMPILAN" KB6 or "EMPILAN" KB12. Disperse with stirring.
- vii) Add "EMPIGEN" BB.
- viii) Add mineral and disperse with stirring.
- ix) Add "EMPICOL" 0251 70J and disperse with stirring.
- x) Add additional nonionic surfactant to clear (if necessary).
- xi) Cool to 40°C.
- xii) Add evaporated water
- xiii) Adjust pH and offload.



**Example 7**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	9	9
DOW CORNING DC 556 SILICONE FLUID (100 %)	9	9
"EMPICOL" 0251 70 J (70 %)	12	17.2
"EMPICOL" CED5 FL (100 %)	5	5
"EMPILAN" KB2 (100 %)	3.5	3.5
"EMPILAN" KB12 (100 %)	3.5	3.5
"EMPIGEN" BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

The formulation forms a microemulsion at 60<sup>0</sup>C and forms a gel when cooled to ambient temperature.

### Example 8

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
HEAVY MINERAL OIL ("KRISTOL" ® M70) (100 %)	15	15
"CERAPHYL" ® GA-D (100 %)	5	5
"EMPICOL" 0251 70 J (70 %)	12	17.2
"EMPICOL" CED5 FL (100 %)	5	5
"EMPILAN" KBE2 (100 %)	3.0	3.0
"EMPILAN" KB12 (100 %)	4.5	4.5
"EMPIGEN" BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

A hazy/opaque emulsion is formed at 60°C and cools to form a clear 'ringing' gel at ambient temperature.

### Physical Data

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for examples 7 and 8**

- i) Charge water and heat to 60°C.
- ii) Add EDTA, sodium benzoate, citric acid and NaOH. Dissolve with stirring.
- iii) Add "EMPICOL" CED5 FL and mix thoroughly.
- iv) Add glycerol.
- v) Add NaCl and disperse with stirring.
- vi) Add "EMPILAN" KBE2 and "EMPILAN" KB12. Disperse with stirring.
- vii) Add "EMPIGEN" BB.
- viii) Blend 50/50 oil phase - oil and cosmetic ingredient. Add to aqueous surfactant solution. Disperse with stirring to form homogeneous emulsion.
- ix) Add "EMPICOL" 0251 70J and disperse.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Adjust pH and offload.

If gel is opaque, re-heat and add additional nonionic surfactant or water.

**Example 10**

<b>Component</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 818 (100 %)	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Forms a microemulsion at 60°C and a 'ringing' gel is obtained after cooling.

**Example 11**

<b>Component</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 840	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

### Physical Data

Density @ 20°C	: 1.0 +/- 0.1	pH (10 %)	: 5.5 +/- 0.5
Appearance:	Clear or hazy gel	Odour	: Characteristic
Set point (typical):	35 +/- 5°C	Viscosity @ 20°C:	N/A

### Method for examples 9, 10 and 11

- i) Blend 50/50 oil phase – oil and cosmetic ingredient. Heat to 60°C.
- ii) Add glycerol and stir to disperse.
- iii) Add “EMPILAN” KBE2 and “EMPILAN” KB12. Disperse with stirring.
- iv) Add “EMPICOL” CED5 FL.
- v) Add “EMPIGEN” BB.
- vi) Add “EMPICOL” 0251 70J.
- vii) Add EDTA, citric acid, sodium benzoate and NaCl. Disperse with stirring.
- viii) Add water.
- ix) Add NaOH.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Adjust pH and offload.

### Example 12

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
EMOLLIENT – FATTY ACID ESTER (100 %)	20	20
“EMPICOL” ® 0251 70 J (70 %)	12	17.2
“EMPICOL” ® CED5 FL (100 %)	5	5
“EMPILAN” ® KB6 (100 %)	5	5
“EMPIGEN” ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Clear gels have been prepared using the following fatty acid esters:

Isopropyl laurate (“ESTOL” ® IPL 1505)  
 Isopropyl myristate (“ESTOL” ® IPM 1512)  
 Isopropyl palmitate (“ESTOL” ® IPP 1517)  
 Isopropyl isostearate (“SCHERCOMOL” ® 318)

### Physical Data

Density @ 20 <sup>0</sup> C	: 1.0 +/- 0.1	pH (10 %)	: 5.5 +/- 0.5
Appearance: Clear or hazy gel		Odour	: Characteristic
Set point (typical): 35 +/- 5 <sup>0</sup> C		Viscosity @ 20 <sup>0</sup> C:	N/A

### Method for example 12

- i) Heat oil phase to 60<sup>0</sup>C.
- ii) Add “EMPILAN” KB6 and stir to disperse.
- iii) Add glycerol and stir to disperse.
- iv) Add “EMPIGEN” BB.
- v) Add “EMPICOL” CED5 FL.

- vi) Add "EMPICOL" 0251 70J.
- vii) Add EDTA, NaCl, sodium benzoate and citric acid. Stir to disperse.
- viii) Add water.
- ix) Add NaOH.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Check pH (10 %).
- xiii) Adjust pH and offload.

The products in each case exhibited cubic symmetry and formed clear microemulsions or dilution with water or heating. The registered trade marks noted above have the following significance:-

"EMPICOL" CVH is a C<sub>8</sub> alkyl ether carboxylic acid

"EMPICOL" LB40 is a C<sub>8</sub> C<sub>10</sub> alkyl sulphate

"EMPICOL" 0251/70J is a C<sub>12-14</sub> alkyl 3 mole ethoxy sulphate

"EMPICOL" 9758 is a C<sub>10</sub> alkyl sulphate

"EMPICOL" CED 5FL is lauryl 6 mole ethoxy carboxylic acid

"EMPIGEN" BB is a C<sub>12-14</sub> alkyl betaine

"EMPIGEN" CDL is coconut amphoteric acetate

"EMPILAN" KB2 is a C<sub>12-14</sub> alkyl 2 mole ethoxylate

"EMPILAN" KB6 is a C<sub>12-14</sub> alkyl 6 mole ethoxylate

"EMPILAN" KB12 is a C<sub>12-14</sub> alkyl 12 mole ethoxylate

"GLUCAPON" 215CS is a C<sub>8-10</sub> alkyl polyclucoside D.P. 1.5

"KATHON" CG is a proprietary biocide

"DOW CORNING" DC556 is phenyl trimethicone

"CERAPHYL" GA-D is maleated soya bean oil

"MIGLYOL" 810/812S is capric/caprylic triglyceride

"MIGLYOL" is capric/caprylic/linoleic triglyceride

"MIGLYOL" 840 is dipropylene glycol dicaprylate/dicaprate

CLAIMS

1. A concentrated personal cleansing composition comprising, by weight of the composition, at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.
2. A composition according to claim 1 wherein the total surfactant has a mean HLB between 10 and 15.
3. A composition according to claim 1 wherein said oil comprises a mineral, fatty ester, glyceride, terpene or silicone oil
4. A composition according to either of claims 1 and 3 wherein the oil comprises at least 16% based on the weight of oil, of a mineral oil.
5. A method for preparing a composition according to claim 1 comprising : (i) forming a mixture (a) of said oil and said oil soluble surfactant; (ii) mixing said mixture (a) with a mixture (b) of said water soluble surfactant and sufficient water to form a lamellar phase with said water soluble surfactant; (iii) maintaining said mixture of (a) and (b) above the  $I_1/L_1$  transition temperature of said composition while diluting said mixture of (a) and (b) with water to form said composition; and (iv) cooling said composition below the  $I_1/L_1$  transition temperature.



**PATENTS ACT 1977**

**PRELIMINARY SPECIFICATION  
(Description)**

**PERSONAL CARE FORMULATIONS**

**Applicant :** **ALBRIGHT & WILSON UK LIMITED**

**Inventors :**

### PERSONAL CARE FORMULATIONS

The present invention relates to shampoo or cleaning compositions suitable for personal care applications in the form of  $I_1$  mesophase systems containing dispersed oil.

Dispersing oil in aqueous shampoo and body wash formulations has presented problems. To prevent the oil phase separating it must either be: (A) emulsified which involves dispersing the oil as colloidal single droplets; (B) microemulsified which involves forming a micellar solution with oil incorporated into surfactant micelles; (C) suspended in a structured surfactant system which typically comprises a dispersion of a surfactant mesophase in aqueous electrolyte; or (D) incorporated into a water soluble solid, pasty or gelatinous composition.

With the exception of microemulsions which are clear, thermodynamically stable, micellar solutions, the foregoing systems are necessarily opaque and contain the oil dispersed in a relatively coarse form, which does not deposit satisfactorily on skin or hair.

However microemulsions are difficult to formulate using the surfactants which are most effective in body wash and other personal care formulations and contain relatively low concentrations of surfactant.

We have now discovered that oil may be stably incorporated into the structure of an  $I_1$  phase to form a clear gel-like composition which contains higher concentrations of surfactant and oil than conventional microemulsions, but, which dissolves in water to form a microemulsion. The novel oil-in- $I_1$  compositions also form microemulsions on heating.

Surfactants are known to form mesophases or liquid crystal phases at concentrations above approximately 30% by weight based on the weight of water and surfactant. Mesophases are phases which exhibit a degree of order intermediate between typical liquids and solids. Generally mesophases combine long range order associated with crystals, with fast molecular motion common to liquids.

The formation of detergent mesophases is well documented. Different surfactants and surfactant mixtures differ widely in their ability to form the numerous different mesophases, and in respect of the conditions of concentration and temperature at which they are formed. For a typical surfactant of the type normally used in cleaning products the following mesophases are usually observed. The concentrations given are illustrative only and may vary considerably from one surfactant or surfactant mixture to the next.

Below approximately 30% surfactant an isotropic  $L_1$  phase is formed (with micelles of surfactant in water). Above 30% surfactant many detergents form a M phase which is not normally used in personal care applications since it does not show suitable flow characteristics and is difficult to dissolve or disperse in water. Above the concentrations required to form an M phase, but usually at concentrations of less than 80% active surfactant, i.e. 60%-80% a G-phase is formed. At concentrations higher than those required to form a G-phase, i.e. typically greater than 80% active surfactant, most surfactants form a hydrated solid, and some, especially non-ionic surfactants form a liquid phase containing dispersed micelle sized droplets of water - an inverted micellar solution known as an  $L_2$  phase.  $L_2$  detergent systems do not disperse readily in water and have a tendency to form undesirable gels, e.g. M phases, on dilution.

Some surfactants form viscous isotropic or VI phases. These are immobile phases usually with a vitreous appearance, and have been relatively little studied compared to the other phases discussed above. They have been virtually ignored in the context of formulating cleaning compositions because most of the surfactants and surfactant systems which are commonly used in cleaning compositions do not form VI phases, at least at normal temperatures, or form them only within narrow concentration ranges and because their known properties as immobile gels has deterred formulators from investigating them. They are recognised as being the most viscous of the lyotropic mesophases.

The different surfactant phases can be recognised by a combination of appearance, rheology, textures under the microscope, electron microscopy and x-ray diffraction or neutron scattering. A detailed description, with illustrations, of the difference textures observable using a polarising microscope, is to be found in the paper by Rosevear JAOCS Vol 31, p628.

The following terms may require explanation or definition:

The "hydrophilic: lipophilic balance", or "HLB" value is used as a measure of the relative affinities of the surfactants for water and oil respectively and correlates with their effectiveness as emulsifiers. HLB value can easily be calculated for alcohol ethoxylates since it is one fifth of the weight percent of ethylene oxide based on the total mole weight. Other surfactants can be assigned equivalent values by applying more complicated formulae or by measuring their relative affinity for water and oil. An HLB value of 20 represents a completely water soluble oil insoluble surfactant, while an HLB value of 0 represents a completely oil soluble and water insoluble surfactant.

"Optically isotropic" surfactant phases do not normally tend to rotate the plane of polarisation of plane polarised light. If a drop of sample is placed between two sheets of optically plane polarising material whose planes are at right angles, and light is shone on to one sheet, optically isotropic surfactant samples do not appear substantially brighter than their surrounding when viewed through the other sheet. Optically anisotropic materials appear substantially brighter. Optically anisotropic mesophases typically show characteristic textures when viewed through a microscope between crossed polarisers, whereas optically isotropic phases usually show a featureless continuum.

"Newtonian liquids" have a viscosity which remains constant at different shear rates. For the purpose of this specification, liquids are considered Newtonian if the viscosity does not vary substantially at shear rates up to  $1000 \text{ sec}^{-1}$ .

"Lamellar" phases are phases which comprise a plurality of bilayers of surfactant arranged in parallel and separated by liquid medium. They include both solid phases and the typical form of the liquid crystal G-phase. G-phases are typically pourable, non-Newtonian, anisotropic products. They are typically viscous-looking, opalescent materials with a characteristic "smeary" appearance on flowing. They form characteristic texture under the polarising microscope and freeze fractured samples have a lamellar appearance under the electron microscope. X-ray diffraction or neutron scattering similarly reveal a lamellar structure, with a principal peak typically between 4 and 10nm, usually 5 to 6nm. Higher order peaks, when present occur at double or higher integral multiples of the Q value of the principal peak. Q is the momentum transfer vector and is related, in the case of lamellar phases, to the repeat spacing d by the equation  $Q = \frac{2n}{d} [\pi]$  where n is the order of the peak.

G-phases, however, can exist in several different forms, including domains of parallel sheets which constitute the bulk of the typical G-phases described above and spherulites formed from a number of concentric spheroidal shells, each of which is a bilayer of surfactant. In this specification the term "lamellar" will be reserved for compositions which are at least partly of the former type. Opaque compositions at least predominantly of the latter type in which the continuous phase is a substantially isotropic solution containing dispersed spherulites are referred to herein as "G-phase compositions". G-phases are sometimes referred to in the literature as  $L_{(\alpha)}$  phases.

$L_1$ -phases are mobile, optically isotropic, and typically Newtonian liquids which show no texture under the polarising microscope. Electron microscopy is capable of resolving the texture of such phases only at very high magnifications, and X-ray or neutron scattering normally gives only a single broad peak typical of a liquid structure, at very small angles close to the reference beam. The viscosity of an  $L_1$ -phase is usually low, but may rise significantly as the concentration approaches the upper phase boundary.

"M-phases" are typically immobile, anisotropic products resembling low melting point waxes. They give characteristic textures under the polarising microscope, and a hexagonal diffraction pattern by X-ray or neutron diffraction which comprises a major peak, usually at values corresponding to a repeat spacing between 4 and 10nm, and sometimes higher order peaks, the first at a Q-value which is  $3^{0.5}$  times the Q-value of the principal peak and the next double the Q-value of the principal peak. M-phases are sometimes referred to in the literature as H-phases.

The viscous isotropic or "VI" phases are typically immobile, non-Newtonian, optically isotropic and are typically transparent, at least when pure. VI phases have a cubic symmetrical diffraction pattern, under X-ray diffraction or neutron scattering, with a principal peak and higher order peaks at  $2^{0.5}$  and  $3^{0.5}$  times the Q-value of the principal peak.

These cubic liquid crystalline phases are sometimes observed immediately following the micellar phase at ambient temperature as the concentration of surfactant is increased. It has been proposed that such VI phases, sometimes referred to as  $I_1$  phase, may arise from the packing of micelles (probably spherical) in a cubic lattice. At ambient temperature a further increase in surfactant concentration usually results in hexagonal phase ( $M_1$ ), which may be followed by a lamellar phase (G).  $I_1$  phases, when they occur, are usually only observed over a narrow range of concentrations, typically just above those at which the  $L_1$ -phase is formed. The location of such VI phases in a phase diagram suggests that the phase is built up of small closed surfactant aggregates in a water continuum.

An inverse form of the  $I_1$  phase (the  $I_2$  phase) has also been reported, possibly between the inverse hexagonal ( $M_2$ ) and  $L_2$  phases. It consists of a surfactant continuum containing a cubic array of inverted micelles. An alternative form of the VI phase called the  $V_1$  phase has been observed at concentrations between the M and G phases and may comprise a bicontinuous system. This may exhibit an even higher viscosity than the  $I_1$ . An inverse phase, the  $V_2$  phase, between the G and  $M_2$  phases has also been postulated.

VI phases are typically examples of "ringing gels". When a jar or beaker containing such a phase is sharply struck, a distinctive vibration can be felt in the composition.

The  $I_1/L_1$  transition temperature will be referred to herein as the melting point of the  $I_1$  phase for convenience, although it is not strictly speaking the melting point since the VI phases are not solids.

All references herein to the formation or existence of specific phases or structures are to be construed, unless the context requires otherwise, as references to their formation or existence at 20°C.

Hexagonal gels (M-phase) have been referred to in the prior art as cleaning compositions, e.g. GB 2 179 055, EP I 153 837 and colloidal gels formed with gelling agents such as synthetic polymers or gelatin have also been suggested, e.g. US 4 465 663.

However these compositions cannot be readily dissolved in water to form microemulsions. They are moreover usually opaque and of an unattractive appearance and often require the presence of solvents such as glycols which add to the cost and are environmentally undesirable.

The use of a type of ringing gel to suspend oil for cosmetic or pharmaceutical applications was described in US 4 026 818 but the formulation requires the presence of hydroxylic solvents and utilises a surfactant system which is unsuitable for shampoo applications. EP O 598 335 describes the use of various cubic phases including  $I_1$  phases as laundry prespotters and for other cleaning formulations. It does not suggest how such phases could be used to suspend oil or form microemulsions. Normally attempts to suspend oil in surfactant mesophases result in coarse droplets of oil being suspended in the aqueous phase of a structured surfactant.

Our invention provides a concentrated personal cleansing composition comprising at least 20% water, 10 to 40% total surfactant and 2 to 40% of a mineral, glyceride, terpene or silicone oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.



The surfactants are preferably selected to provide an I<sub>1</sub> phase over a comparatively broad surfactant concentration range e.g. more than  $\pm 5\%$  or greater, which range typically lies above 15% by weight total surfactant based on the weight of the composition e.g. between 20% and 40% by weight surfactant usually between 25% and 60%.

The surfactants are preferably selected to provide an I<sub>1</sub> phase which melts above 30°C e.g. above 35°C, most preferably above 40°C. Preferably the I<sub>1</sub> phase melts at a temperature substantially below 100°C, e.g. below 90°C, more preferably below 80°C, most preferably below 70°C, especially below 60°C, typically below 55°C, usually below 50°C.

The surfactant mixture preferably has a mean HLB based on the molar proportions of the components between 10 and 15 e.g. 11 to 14. The surfactants preferably comprise non-ionic surfactants such as ethoxylated alcohols. It has been found that highly ethoxylated fatty alcohols, e.g. more than 10 EO groups, preferably more than 15 EO groups, especially 18 to 50 EO groups form I<sub>1</sub> phases particularly readily.

Other non-ionic surfactants which may be present include:-

alkyl phenol ethoxylates, fatty acid ethoxylates, fatty acid monoalkylolamide ethoxylates, fatty alcohol propoxylates, fatty anime alkoxyates and fatty acid glyceryl ester ethoxylates. Other non-ionic compounds suitable for inclusion in compositions of the present invention include mixed ethylene oxide propylene oxide alkoxyates, low relative molecular mass polyethylene glycols e.g. PEG600 and PEG200, ethylene glycol monoesters, amine oxides and alkyl polyglycosides, alkyl sugar esters including alkyl sucrose esters and alkyl oligosaccharide ester, alkyl capped polyvinyl alcohol and alkyl capped polyvinyl pyrrolidone.

Compositions of the invention may also comprise anionic surfactants, in addition to or instead of non-ionic surfactants. Anionic surfactant may comprise a  $C_{10-20}$  alkyl benzene sulphonate or an alkyl ether sulphate which is preferably the product obtained by ethoxylating a natural fatty or synthetic  $C_{10-20}$  e.g. a  $C_{12-14}$  alcohol with from 1 to 20, preferably 2 to 10 e.g. 3 to 4 ethyleneoxy groups, optionally stripping any unreacted alcohol, reacting the ethoxylated product with a sulphating agent and neutralising the resulting alkyl ether sulphuric acid with a base. The term also includes alkyl glyceryl sulphates, and random or block copolymerised alkyl ethoxy/propoxy sulphates.

The anionic surfactant may also comprise, for example,  $C_{10-20}$  e.g.  $C_{12-18}$  alkyl sulphate.

The surfactant may comprise a  $C_{8-20}$  e.g.  $C_{10-20}$  aliphatic soap. The soap may be saturated or unsaturated, straight or branched chain.

Preferred examples include dodecanoates, myristates, stearates, oleates, linoleates, linoleates and palmitates and coconut and tallow soaps.

The surfactant may include other anionic surfactants, such as olefin sulphonates, paraffin sulphonates, taurides, isethionates, ether sulphonates, ether carboxylates, aliphatic ester sulphonates e.g. alkyl glyceryl sulphonates, sulphosuccinates or sulphosuccinamates.

The cation of any anionic surfactant is typically sodium but may alternatively be potassium, lithium, calcium, magnesium, ammonium, or an alkyl ammonium having up to 6 aliphatic carbon atoms including isopropyl ammonium, monoethanol ammonium, diethanol ammonium, and triethanol ammonium.

Ammonium and ethanol ammonium salts are generally more soluble than the sodium salts. Mixtures of the above cations may be used.

The composition may contain amphoteric surfactants such as betaines sulphobetaines, amido betaines or imidazoline betaines.

The I<sub>1</sub> phase may be conveniently prepared by mixing the oil and oil soluble surfactant and adding sufficient water to the water soluble surfactant to maintain a lamellar phase. The oil and oil soluble surfactant may be stirred into the lamellar composition at elevated temperature, above the melting point of the desired I<sub>1</sub> phase. The composition is then diluted with hot water until a microemulsion is formed and then cooled to solidify it into the I<sub>1</sub> phase.

The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether) or a fatty glyceride, a terpene oil such as limonene or a silicone oil. Mixtures of oils may be used. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil. Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolved in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds, ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide may be dispersed in the hot microemulsion prior to cooling. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may be suspended. Other active ingredients which may be suspended include insect repellants and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

Electrolytes tend to break  $I_1$  phase structure and are preferably present in concentrations below 10% based on total weight of the compositions, more preferably below 5%, e.g. 0 to 3%, most preferably 0 to 1%. Generally we prefer that electrolyte be substantially absent. Adventitious chloride or sulphate present as impurities in the surfactant can be tolerated. Small amounts of builder such as citrates, pyrophosphates, polyphosphates may optionally be included.

Water soluble solvents are generally undesirable and are not required to form stable  $I_1$  structures according to the invention. We therefore prefer that they should be substantially absent. Although small amounts of, for example, ethanol or propanol may sometimes be desired for special purposes, they are preferably present in amounts less than 5% by weight, more preferably less than 3% by weight, most preferably less than 2% by weight, e.g. less than 1% by weight.

The composition may optionally contain hydrotropes such as sodium lower alkyl benzene sulphonate e.g. sodium toluene, xylene or cumene sulphonate or urea, however these are not generally necessary and are not generally preferred. We prefer that these should be present in quantities less than 5% by weight, more preferably less than 4%, especially less than 2% e.g. 0 to 1%. They may be useful occasionally to avoid haziness of the gel.

The total amount of water is preferably from 25 to 60% by weight of the composition, more preferably 30 to 50%, e.g. 35 to 50%. The total weight percentage of surfactant based on the weight of the composition is preferably from 15 to 35%, e.g. 20 to 30%. The proportion of oil is preferably greater than 5%, more preferably greater than 8%, e.g. 10 to 30%, especially 15 to 25% by weight based on the weight of the composition. The oil soluble surfactant is preferably present in a proportion of more than 1:5 based on the weight of oil, more preferably from 1:2 to 5:1. The oil soluble surfactant preferably has an HLB of from 3 to 9 e.g. 4 to 8.

The weight ratio of water soluble surfactant to oil soluble surfactant is preferably 1:1 to 30:1, more preferably 2:1 to 20:1, typically 3:1 to 15:1, e.g. 4:1 to 10:1. The water soluble surfactant preferably has an HLB greater than 12, more preferably greater than 13, especially 14 to 19.

The product may be cast into shaped bodies or formed into particles or granules, e.g. by spray cooling a hot solution of the  $L_1$  phase formed on melting the composition.

The composition may be converted into a microemulsion phase by addition of water, by heating above the melting point or by adding electrolyte such as salt and the invention includes  $L_1$  phases when so prepared.

The invention will be illustrated by the following examples:

**Example 1**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	20	20
"EMPICOL"® 0251/70J (70%)	11.2	16
"EMPIGEN"® BB (30%)	4.8	16
"GLUCAPON"® 215 CS UP (65%)	6	9.2
"EMPILAN"® KB2 (100%)	7.5	7.5
SODIUM CHLORIDE (100%)	2	2
PERFUME (100%)	0.5	0.5
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.1	0.1
CITRIC ACID (100%)	0.2	0.2
BENZOIC ACID (100%)	0.3	0.3
SODIUM HYDROXIDE (47%)	0.1	0.2
WATER	---	Balance

The method of mixing comprised the following steps:-

1. Charge 50% of water
2. Heat to 60°C
3. Add EDTA, sodium benzoate, citric acid and 47% NaOH dissolve with stirring
4. Add "EMPIGEN" BB
5. Add mineral oil and disperse with stirring
6. Add "EMPILAN" KB 2 and mix thoroughly
7. Add "EMPICOL" 0251/70j
8. Add remaining water
9. Add "GLUCAPON" 215 CS UP
10. Add further KB 2 until clear
11. Cool
12. Add evaporated water
13. Adjust pH

**Physical Data**

pH (10%)	: 5.5 ± 0.1	Density @ 20°C	: 1.0 ± 0.1 g cm <sup>-3</sup>
Solids (%)	: ~ 53% (typical)	Appearance	: Clear or Hazy Gel
Odour	: Characteristic	Set Point (typical)	: 30°C
Viscosity @ 20°C : N/A			

The product was examined by x-ray diffraction and exhibited peaks at 13.145nm (intense and sharp), 7.943nm (ill defined) and 6.355nm (small), indicating cubic symmetry, and formed a clear microemulsion on dilution or heating. The latter gave good even distribution of oil applied to skin.

## **Example 2**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>w/w (%)</u></b>
MINERAL OIL (100%)	15	15
"EMPICOL"® CDL30J/35 (22%)	8	35.4
"EMPIGEN"® BB (30%)	8	26.7
"EMPICOL"® 0785 (40%)	2	5
"EMPILAN"® KB2 (100%)	6	6
"EMPILAN"® KB6 (100%)	6	6
CITRIC ACID (100%)	0.5	0.5
PERFUME (100%)	0.2	0.2
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.2	0.2
"KATHON"®	---	0.2
WATER	---	Balance
<b>TOTAL</b>	<b>45.8</b>	<b>100</b>

### **Physical Data**

Appearance	: Clear Liquid/Gel	Odour	: Characteristic Odour
Solids	: 36.5% (typical)	pH (100%)	: 5.5 - 6.5 (typical)
Odour	: Characteristic	Set Point	: 20 ± 5°C

Viscosity (Carrimed Rheometer @ 20°C : N/A

The product had small angle x-ray diffraction peaks characteristic of cubic symmetry and formed a clear microemulsion on dilution with water or warming. The latter gave good even deposition of oil on skin.

**Examples 3 and 4**

The following ingredients were mixed at 60°C and cooled to form ringing gels:

Component	1		2	
	Solids (%)	w/w (%)	Solids (%)	w/w (%)
"EMPIGEN"® CDL30J/35 (22%)	8	36.4	8	36.4
"EMPIGEN"® BB (30%)	8	26.7	8	26.7
"EMPICOL"® LB40 (40%)	4	7.5	3	7.5
"EMPICOL"® CVH (90%)	4	4	---	---
"EMPILAN"® KB2 (100%)	5.5	5.5	6	6
TRIETHANOLAMINE (100%)	1.1	1.1	---	---
CITRIC ACID	1	0.75	0.75	0.75
ETHYLENE DIAMINE				
TETRACETIC ACID	0.05	0.05	0.05	0.05
"KATHON"® CG (100%)	0.05	0.05	0.05	0.05
LIGHT MINERAL (100%)	14	14	20	20
WATER	---	Balance	---	Balance
<b>TOTAL</b>	<b>45.7</b>	<b>100</b>	<b>46.1</b>	<b>100</b>
<b>Appearance</b>	Clear Gel		Clear Gel	



The products in each case exhibited cubic symmetry and formed clear microemulsions or dilution with water or heating. The registered trade marks noted above have the following significance:-

"EMPICOL" CVH is a C<sub>8</sub> alkyl ether carboxylic acid

"EMPICOL" LB40 is a C<sub>8</sub> C<sub>10</sub> alkyl sulphate

"EMPICOL" 0251/70J is a C<sub>12-14</sub> alkyl 3 mole ethoxy sulphate

"EMPICOL" 9758 is a C<sub>10</sub> alkyl sulphate

"EMPIGEN" BB is a C<sub>12-14</sub> alkyl betaine

"EMPIGEN" CDL is coconut amphi acetate

"EMPILAN" KB2 is a C<sub>12-14</sub> alkyl 2 mole ethoxylate

"EMPILAN" KB6 is a C<sub>12-14</sub> alkyl 6 mole ethoxylate

"GLUCAPON" 215CS is a C<sub>8-10</sub> alkyl polyclucoside D.P. 1.5

"KATHON" CG is a proprietary biocide

# Exhibit 4

# HUNTSMAN

Huntsman Surface Sciences UK Limited

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Facsimile +44 (0) 121 420 5700

Date **December 05, 2001**  
Direct Tel **+44 (0) 121 420 5431**  
Direct Fax **+44 (0) 121 420 5437**  
Your Ref **00945731.8**  
Our Ref **MPD315/EP/PCT/  
RGMS/SMSL1**

**VIA FAX -  
CONFIRMATION BY COURIER**

European Patent Office  
Erhardtstrasse 27  
D-80298 Munich  
GERMANY

Dear Sirs

**ENTRY INTO REGIONAL PHASE  
EUROPEAN PATENT APPLICATION NO. 00945731.8  
APPLICANT : RHODIA CONSUMER SPECIALTIES LIMITED AND  
JOHNSON & JOHNSON CONSUMER COMPANIES INC  
ASSIGNED TO HUNTSMAN INTERNATIONAL LLC**

We file herewith the following document in respect of entry into the regional  
stand before the EPO under Chapter II PCT. The 30 month period from priority  
expires on 10 December 2001.

Form 1200

Voucher for payment of fees from our EPO Deposit Account  
Number : 28 05 03 16 and comprising

	<u>DEM</u>
Filing Fee	248.39
Designation Fees (18 x DEM 148.64)	2675.52
50% Examination Fee	1399.40
Claims Fee	<u>78.23</u>
	4401.54
	=====

Contd/.....

Huntsman Surface Sciences UK Limited

Place of Registration : England. Registered Number : 4056146. Registered Office : Haverton Hill Road, Billingham, TS23 1PS

European Patent Office  
Munich

December 05, 2001

We would advise that this Patent Application was part of the assets, which has now been sold to HUNTSMAN INTERNATIONAL LLC, a company incorporated in the state of Delaware had having its principal place of business at 500 Huntsman Way, Salt Lake City, Utah 84108, USA, and this was advised to you in our letter of August 14, 2001 (a copy of which is enclosed).

If you require any further information with regard to the above please do not hesitate to contact this office.

Yours faithfully  
For and on behalf of  
Huntsman Surface Sciences UK Limited



**R G M SAVIDGE**  
Authorised Representative (GA43702)

Encs

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**Eintritt in die regionale  
Phase vor dem EPA  
als Bestimmungsamt  
oder ausgewähltem Amt**

**Entry into the regional  
phase before the EPO  
as designated or elected  
Office**

**Entrée dans la phase  
régionale devant l'OEB  
agissant en qualité d'office  
désigné ou élu**

Europäische Anmeldenummer oder, falls  
nicht bekannt, PCT-Aktenzeichen oder  
PCT-Veröffentlichungsnummer

European application number, or, if not  
known, PCT application or publication  
number

Numéro de dépôt de la demande de  
brevet européen ou, à défaut, numéro  
de dépôt PCT ou de publication PCT

00945731.8

Zeichen des Anmelders oder Vertreters  
(max. 15 Positionen)

Applicant's or representative's reference  
(max. 15 spaces)

Référence du demandeur ou du mandataire  
(15 caractères ou espaces au maximum)

MPD315/PCT/EP



**1. Anmelder**  
Die Angaben über den (die)  
Anmelder sind in der internationalen  
Veröffentlichung enthalten oder vom  
Internationalen Büro nach der  
internationalen Veröffentlichung  
vermerkt worden.



Änderungen, die das Internationale  
Büro noch nicht vermerkt hat, sind  
auf einem Zusatzblatt angegeben.

**Zustellanschrift**  
(siehe Merkblatt II, 1)

**1. Applicant**  
Indications concerning the  
applicant(s) are contained in the  
international publication or recorded  
by the International Bureau after the  
international publication.

Changes which have not yet been  
recorded by the International Bureau  
are set out on an additional sheet.

**Address for correspondence**  
(see Notes II, 1)

AS IN 2

**1. Demandeur**  
Les indications concernant le(s) de-  
mandeur(s) figurent dans la publica-  
tion internationale ou ont été enregistrées  
par le Bureau international après la  
publication internationale.

Les changements qui n'ont pas encore  
été enregistrés par le Bureau inter-  
national sont indiqués sur une feuille  
additionnelle.

**Adresse pour la correspondance**  
(voir notice II, 1)

**2. Vertreter**

Name (Nur einen Vertreter angeben,  
der in das europäische Patentregister  
eingetragen und an den zugestellt  
wird)

**Geschäftsanschrift**

**Telefon**

**Telefax**      **Telex**



Weiterer Vertreter auf Zusatzblatt

**2. Representative**

Name (Name only one  
representative who will be listed in  
the Register of European Patents and  
to whom notification will be made)

**Address of place of business**

**Telephone**

+44 121 420 5430

**Fax**      **Telex**

+44 121 420 5437

Additional representative(s) on  
additional sheet

**2. Mandataire**

Nom (N'indiquer qu'un seul  
mandataire, qui sera inscrit au  
Registre européen des brevets et  
auquel signification sera faite)

**Adresse professionnelle**

**Téléphone**

**Téléfax**      **Telex**

Autre(s) mandataire(s) sur une feuille  
additionnelle

**3. Vollmacht**



Einzelvollmacht ist beigelegt.



Allgemeine Vollmacht ist registriert  
unter Nummer:



Allgemeine Vollmacht ist eingereicht,  
aber noch nicht registriert.



Die beim EPA als PCT-Anmeldeamt  
eingereichte Vollmacht schließt  
ausschließlich die regionale Phase ein.

**3. Authorisation**

Individual authorisation is attached.

General authorisation has been  
registered under No:

GA43702

A general authorisation has been  
filed, but not yet registered.

The authorisation filed with the EPO  
as PCT receiving Office expressly  
includes the regional phase.

**3. Pouvoir**

Un pouvoir spécial est joint.

Un pouvoir général a été enregistré  
sous le n°:

Un pouvoir général a été déposé  
mais n'est pas encore enregistré

Le pouvoir général déposé à l'OEB  
agissant en qualité d'office réceptif  
au titre du PCT s'applique expressé-  
ment à la phase régionale.

<p>4. <b>Prüfungsantrag</b> Hiermit wird die Prüfung der Anmeldung gemäß Art. 94 EPU beantragt. Die Prüfungsgebühr wird (wurde) entrichtet.</p>	<p>4. <b>Request for examination</b> Examination of the application under Art. 94 EPC is hereby requested. The examination fee is being (has been, will be) paid.</p>	<p>4. <b>Requête en examen</b> Il est demandé que soit examinée la demande de brevet conformément à l'art. 94 CBE. Il est (a été, sera) procédé au paiement de la taxe d'examen.</p>
<p><i>Prüfungsantrag in einer zugelassenen Nichtamtssprache (siehe Merkblatt III, 6.2):</i></p>	<p><i>Request for examination in an admissible non-EPC language (see Notes III, 6.2)</i></p>	<p><i>Requête en examen dans une langue non officielle autorisée (voir notice II, 6.2):</i></p>
<p>5. <b>Abschriften</b> Zusätzliche Abschrift(en) der im ergänzenden europäischen Recherchenbericht angeführten Schriftstücke wird (werden) beantragt.</p> <p>Anzahl der zusätzlichen Sätze von Abschriften</p>	<p>5. <b>Copies</b> Additional copy (copies) of the documents cited in the supplementary European search report is (are) requested.</p> <p>Number of additional sets of copies</p>	<p>5. <b>Copies</b> Prière de fournir une ou plusieurs copie supplémentaire des documents cités dans le rapport complémentaire de recherche européenne.</p> <p>Nombre de jeux supplémentaires de copies</p>
<p>6. <b>Für das Verfahren vor dem EPA bestimmte Unterlagen</b></p>	<p>6. <b>Documents intended for proceedings before the EPO</b></p>	<p>6. <b>Pièces destinées à la procédure devant l'OEB</b></p>
<p>6.1 Dem Verfahren vor dem EPA als Bestimmungsamt (PCT II) sind folgende Unterlagen zugrunde zu legen:</p>	<p>6.1 Proceedings before the EPO as designated Office (PCT II) are to be based on the following documents:</p>	<p>6.1 La procédure devant l'OEB agissant en qualité d'office désigné (PCT II) se fonde sur les pièces suivantes:</p>
<p><input checked="" type="checkbox"/> die vom Internationalen Büro veröffentlichten Anmeldeunterlagen (mit allen Ansprüchen, Beschreibung und Zeichnungen), gegebenenfalls mit den geänderten Ansprüchen nach Art. 19 PCT</p>	<p>the application documents published by the International Bureau (with all claims, description and drawings), where applicable with amended claims under Art. 19 PCT</p>	<p>les pièces de la demande publiée par le Bureau international (avec toutes les revendications, la description et les dessins), éventuellement avec les revendications modifiées conformément à l'article 19 du PC</p>
<p><input type="checkbox"/> soweit sie nicht ersetzt werden durch die in drei Stücken beigefügten Änderungen.</p>	<p>unless replaced by the amendments enclosed in triplicate.</p>	<p>dans la mesure où elles ne sont pas remplacées par les modifications jointes en trois exemplaires.</p>
<p><i>Falls nötig, sind Klarstellungen auf einem Zusatzblatt einzureichen!</i></p>	<p><i>Where necessary, clarifications must be submitted on a separate sheet!</i></p>	<p><i>Le cas échéant, des explications doivent être jointes sur une feuille additionnelle!</i></p>
<p>6.2 Dem Verfahren vor dem EPA als ausgewähltem Amt (PCT II) sind folgende Unterlagen zugrunde zu legen:</p>	<p>6.2 Proceedings before the EPO as elected Office (PCT II) are to be based on the following documents:</p>	<p>6.2 La procédure devant l'OEB agissant en qualité d'office élu (PCT II) se fonde sur les pièces suivantes:</p>
<p><input checked="" type="checkbox"/> die dem internationalen vorläufigen Prüfungsbericht zugrunde gelegten Unterlagen, einschließlich seiner eventuellen Anlagen <i>(Solche Anlagen müssen immer in drei Stücken beigelegt werden)</i></p>	<p>the documents on which the international preliminary examination report is based, including its possible annexes <i>(Such annexes must always be filed in triplicate)</i></p>	<p>les pièces sur lesquelles se fonde le rapport d'examen préliminaire international, y compris ses annexes éventuelles <i>(De telles annexes sont toujours à joindre en trois exemplaires)</i></p>
<p><input type="checkbox"/> soweit sie nicht ersetzt werden durch die in drei Stücken beigefügten Änderungen.</p>	<p>unless replaced by the amendments enclosed in triplicate.</p>	<p>dans la mesure où elles ne sont pas remplacées par les modifications jointes en trois exemplaires.</p>
<p><i>Falls nötig, sind Klarstellungen auf einem Zusatzblatt einzureichen!</i></p>	<p><i>Where necessary, clarifications must be submitted on a separate sheet!</i></p>	<p><i>Le cas échéant, des explications doivent être jointes sur une feuille additionnelle!</i></p>
<p><input checked="" type="checkbox"/> Sind dem EPA als mit der internationalen vorläufigen Prüfung beauftragten Behörde Versuchsberichte zugegangen, dürfen diese dem Verfahren vor dem EPA zugrunde gelegt werden.</p>	<p>If the EPO as International Preliminary Examining Authority has received test reports, these may be used as the basis of proceedings before the EPO</p>	<p>Si l'OEB, agissant en qualité d'administration chargée de l'examen préliminaire international, a reçu des rapports d'essais, ceux-ci peuvent constituer la base de la procédure devant l'OEB</p>

## 7. Übersetzungen

Beigefügt sind die nachfolgend angekreuzten Übersetzungen in einer der Amtssprachen des EPA (Deutsch, Englisch, Französisch):

- Im Verfahren vor dem EPA als Bestimmungsamt oder ausgewähltem Amt (PCT I + II):

☐ Übersetzung der ursprünglich eingereichten internationalen Anmeldung (Beschreibung, Ansprüche, etwaige Textbestandteile in den Zeichnungen), der veröffentlichten Zusammenfassung, und etwaiger Angaben über Mikroorganismen nach Regel 13<sup>ter</sup>.3 und 13<sup>ter</sup>.4 PCT, in drei Stücken

☐ Übersetzung der prioritätsbegründenden Anmeldung(en), in einem Stück

- Zusätzlich im Verfahren vor dem EPA als Bestimmungsamt (PCT II):

☐ Übersetzung der nach Art. 19 PCT geänderten Ansprüche nebst Erklärung, falls diese dem Verfahren vor dem EPA zugrunde gelegt werden sollen (siehe Feld 6), in drei Stücken

- Zusätzlich im Verfahren vor dem EPA als ausgewähltem Amt (PCT II):

☐ Übersetzung der Anlagen zum internationalen vorläufigen Prüfungsbericht, in drei Stücken

## 7. Translations

Translations in one of the official languages of the EPO (English, French, German) are enclosed as crossed below:

- In proceedings before the EPO as designated or elected Office (PCT I + II):

Translation of the international application (description, claims, any text in the drawings) as originally filed, of the abstract as published and of any indication under Rule 13<sup>ter</sup>.3 and 13<sup>ter</sup>.4 PCT regarding micro-organisms, in triplicate

Translation of the priority application(s), in one copy

- In addition, in proceedings before the EPO as designated Office (PCT II):

Translation of amended claims and any statement under Art. 19 PCT, if the claims as amended are to form the basis for the proceedings before the EPO (see Section 6), in triplicate

- In addition, in proceedings before the EPO as elected Office (PCT III):

Translation of any annexes to the international preliminary examination report, in triplicate

## 7. Traductions

Vous trouverez, ci-joint, les traductions cochées ci-après dans l'une des langues officielles de l'OEB (allemand, anglais, français) :

- Dans la procédure devant l'OEB agissant en qualité d'office désigné ou élu (PCT I + II):

Traduction de la demande internationale telle que déposée initialement (description, revendications, textes figurant éventuellement dans les dessins), de l'abrége publié, et de toutes indications visées aux règles 13<sup>ter</sup>.3 et 13<sup>ter</sup>.4 du PCT concernant les micro-organismes, en trois exemplaires

Traduction de la (des) demande(s) ouvrant le droit de priorité, en un exemplaire

- De plus, dans la procédure devant l'OEB agissant en qualité d'office désigné (PCT II) :

Traduction des revendications modifiées et de la déclaration faite conformément à l'article 19 du PCT si la procédure devant l'OEB doit être fondée sur les revendications modifiées (voir la rubrique 6), en trois exemplaires

- De plus, dans la procédure devant l'OEB agissant en qualité d'office élu (PCT III) :

Traduction des annexes du rapport d'examen préliminaire international, en trois exemplaires

## 8. Biologisches Material

☐ Die Erfindung bezieht sich auf bzw. verwendet biologisches Material, das nach Regel 28 EPU hinterlegt worden ist.

☐ Die Angaben nach Regel 28(1)c) EPU (falls noch nicht bekannt, die Hinterlegungsstelle und das (die) Bezugszeichen (Nummer, Symbole usw.) des Hinterlegers) sind in der internationalen Veröffentlichung oder in der gemäß Feld 7 eingereichten Übersetzung enthalten auf:

Seite(n) / Zeile(n)

Die Empfangsbescheinigung(en) der Hinterlegungsstelle

☐ ist (sind) beigefügt

☐ wird (werden) nachgereicht

☐ Verzicht auf die Verpflichtung des Antragstellers nach Regel 28(3) auf gezeichnetem Schriftstück

## 8. Biological material

The invention relates to and/or uses biological material deposited under Rule 28 EPC

The particulars referred to in Rule 28(1)(c) EPC (if not yet known, the depository institution and the identification reference(s) (number, symbols etc.) of the depositor) are given in the translation submitted under Section 7 on:

page(s) / line(s)

The receipt(s) of deposit issued by the depository institution

is (are) enclosed

will be filed at a later date

Waiver of the right to an undertaking from the requester pursuant to Rule 28(3) attached

## 8. Matière biologique

L'invention concerne et/ou utilise la matière biologique, déposée conformément à la règle 28 CBE.

Les indications visées à la règle 28(1)c) CBE (si pas encore connues l'autorité de dépôt et la (les) référence(s) d'identification (numéros, symboles, etc.) du déposant) figurent dans la publication internationale ou, dans une traduction produite conformément à la rubrique 7 à la / au :

page(s) / ligne(s)

Le(s) récépissé(s) de dépôt délivré(s) par l'autorité de dépôt

est (sont) joint(s)

sera (seront) produit(s) ultérieurement

Renonciation, sur document distinct, à l'engagement du requérant au titre de la règle 28(3)

### 9. Nucleotid- und Aminosäuresequenzen

Die nach Regeln 5.2 und 13<sup>ter</sup> PCT sowie Regel 104b (3a) EPU erforderlichen Unterlagen liegen dem EPA bereits vor.

Das schriftliche Sequenzprotokoll wird anliegend in einer Amtssprache des EPA nachgereicht.

Das Sequenzprotokoll geht nicht über den Inhalt der Anmeldung in der ursprünglich eingereichten Fassung hinaus.

Der vorgeschriebene maschinenlesbare Datenträger ist beigelegt.

Die auf dem Datenträger gespeicherte Information stimmt mit dem schriftlichen Sequenzprotokoll überein.

### 9. Nucleotide and amino acid sequences

The items necessary in accordance with Rules 5.2 and 13<sup>ter</sup> PCT and Rule 104b (3a) EPC have already been furnished to the EPO.

The written sequence listing is furnished herewith in an official language of the EPO.

The sequence listing does not include matter which goes beyond the content of the application as filed.

The prescribed machine-readable data carrier is enclosed.

The information recorded on the data carrier is identical to the written sequence listing.

### 9. Séquences de nucléotides et d'acides aminés

Les pièces requises selon les règles 5.2 et 13<sup>ter</sup> PCT et la règle 104b (3a) CBE ont déjà été déposées auprès de l'OEB.

La liste de séquences écrite est produite conjoint dans une des langues officielles de l'OEB.

La liste de séquences ne contient pas d'éléments s'étendant au-delà du contenu de la demande telle qu'elle a été déposée.

Le support de données prescrit, déchiffirable par machine, est annexé.

L'information figurant sur le support de données est identique à celle qui contient la liste de séquences écrites.

### 10. Benennungsgebühren

10.1 Benennungsgebühren werden für nachstehende in der internationalen Anmeldung bestimmte Vertragsstaaten des EPU entrichtet.

<input checked="" type="checkbox"/>	AT	Osterreich
<input checked="" type="checkbox"/>	BE	Belgien
<input checked="" type="checkbox"/>	CH/LI	Schweiz und Liechtenstein
<input checked="" type="checkbox"/>	CY	Zypern
<input checked="" type="checkbox"/>	DE	Deutschland
<input checked="" type="checkbox"/>	DK	Dänemark
<input checked="" type="checkbox"/>	ES	Spanien
<input checked="" type="checkbox"/>	FI	Finnland
<input checked="" type="checkbox"/>	FR	Frankreich
<input checked="" type="checkbox"/>	GB	Vereinigtes Königreich
<input checked="" type="checkbox"/>	GR	Griechenland
<input checked="" type="checkbox"/>	IE	Irland
<input checked="" type="checkbox"/>	IT	Italien
<input checked="" type="checkbox"/>	LU	Luxemburg
<input checked="" type="checkbox"/>	MC	Monaco
<input checked="" type="checkbox"/>	NL	Niederlande
<input checked="" type="checkbox"/>	PT	Portugal
<input checked="" type="checkbox"/>	SE	Schweden

### 10. Designation fees

10.1 Designation fees are paid in respect of the following EPC Contracting States designated in the international application for a European patent:

<input checked="" type="checkbox"/>	Austria
<input checked="" type="checkbox"/>	Belgium
<input checked="" type="checkbox"/>	Switzerland and Liechtenstein
<input checked="" type="checkbox"/>	Cyprus
<input checked="" type="checkbox"/>	Germany
<input checked="" type="checkbox"/>	Denmark
<input checked="" type="checkbox"/>	Spain
<input checked="" type="checkbox"/>	Finland
<input checked="" type="checkbox"/>	France
<input checked="" type="checkbox"/>	United Kingdom
<input checked="" type="checkbox"/>	Greece
<input checked="" type="checkbox"/>	Ireland
<input checked="" type="checkbox"/>	Italy
<input checked="" type="checkbox"/>	Luxembourg
<input checked="" type="checkbox"/>	Monaco
<input checked="" type="checkbox"/>	Netherlands
<input checked="" type="checkbox"/>	Portugal
<input checked="" type="checkbox"/>	Sweden

### 10. Taxes de désignation

10.1 Les taxes de désignation sont acquittées pour ceux des Etats contractants de la CBE désignés dans la demande internationale qui sont indiqués ci-après:

<input checked="" type="checkbox"/>	Autriche
<input checked="" type="checkbox"/>	Belgique
<input checked="" type="checkbox"/>	Suisse et Liechtenstein
<input checked="" type="checkbox"/>	Chypre
<input checked="" type="checkbox"/>	Allemagne
<input checked="" type="checkbox"/>	Danemark
<input checked="" type="checkbox"/>	Espagne
<input checked="" type="checkbox"/>	Finlande
<input checked="" type="checkbox"/>	France
<input checked="" type="checkbox"/>	Royaume-Uni
<input checked="" type="checkbox"/>	Grèce
<input checked="" type="checkbox"/>	Irlande
<input checked="" type="checkbox"/>	Italie
<input checked="" type="checkbox"/>	Luxembourg
<input checked="" type="checkbox"/>	Monaco
<input checked="" type="checkbox"/>	Pays-Bas
<input checked="" type="checkbox"/>	Portugal
<input checked="" type="checkbox"/>	Suède

10.2 Bisher ist nicht beabsichtigt, Benennungsgebühren für die in Feld 10.1 nicht angekreuzten, aber in der internationalen Anmeldung bestimmten Vertragsstaaten des EPU zu entrichten. Insofern wird auf die Zustellung einer Mitteilung nach Regel 65b(1) EPU verzichtet. Sofern diese Benennungsgebühren nicht bis zum Ablauf der in Regel 65a(2) EPU vorgesehenen Nachfrist entrichtet werden, wird beantragt, von einer Mitteilung nach Regel 69(1) EPU abzusehen.

10.2 At present it is not intended to pay designation fees for the EPC Contracting States not marked with a cross under 10.1 but designated in the international application. No communication under Rule 65a(1) EPC in respect of these designation fees need be notified, if they have not been paid by the time the period of grace allowed in Rule 35a(2) EPC expires. It is requested that no communication be sent under Rule 69(1) EPC.

10.2 Il n'est pas actuellement envisagé d'acquitter les taxes de désignation pour les Etats contractants de la CBE qui ne sont pas cochés sous la rubrique 10.1, mais qui sont désignés dans la demande internationale. Le demandeur renonce ainsi à la notification prévue à la règle 65b(1) CBE. Si des taxes de désignation ne sont pas acquittées à l'expiration du délai supplémentaire prévu à la règle 65b(2) CBE, il est demandé de s'abstenir d'envoyer une notification, établie conformément à la règle 69(1) CBE.

11) Only those designated in the international application on or after 1 April 1993 contribute to the EPO's costs for the EPC Contracting States designated in the international application for a European patent.

11) Only those designated in the international application on or after 1 April 1993 contribute to the EPO's costs for the EPC Contracting States designated in the international application for a European patent.

11) Seuls ceux désignés dans la demande internationale à partir du 1<sup>er</sup> avril 1993 ou après cette date contribuent aux dépenses de l'OEB pour les Etats contractants de la CBE désignés dans la demande internationale pour un brevet européen.



<input checked="" type="checkbox"/> <b>11. Erstreckung des europäischen Patents</b> Diese Anmeldung gilt auch als Erstreckungsantrag hinsichtlich aller in der internationalen Anmeldung bestimmten Nicht-Vertragsstaaten des EPÜ, mit denen bei Einreichung der internationalen Anmeldung "Erstreckungsabkommen" in Kraft waren". Die Erstreckung wird jedoch nur wirksam, wenn die vorgeschriebene Erstreckungsgebühr entrichtet wird. Der Anmelder beabsichtigt, die Erstreckungsgebühr für die nachfolgend angekreuzten Staaten zu entrichten:  <input type="checkbox"/> SI Slowenien (* ab 1. März 1994) <input type="checkbox"/> LT Litauen (* ab 5. Juli 1994) <input type="checkbox"/> LV Lettland (* ab 1. Mai 1995) <input type="checkbox"/> AL Albanien (* ab 1. Februar 1996) <input type="checkbox"/> RO Rumänien (* ab 15. Oktober 1996) <input type="checkbox"/> MK Ehemalige jugoslawische Republik Mazedonien (* ab 1. November 1997)  <input type="checkbox"/> _____ " _____ "	<b>11. Extension of the European patent</b> This application is also considered as being a request for extension to all the non-Contracting States to the EPC designated in the international application with which "extension agreements" were in force on the date of filing the international application". However, the extension only takes effect if the prescribed extension fee is paid. The applicant intends to pay the extension fee for the States marked with a cross below:  Slovenia (* as of 1 March 1994) Lithuania (* as of 5 July 1994) Latvia (* as of 1 May 1995) Albania (* as of 1 February 1996) Romania (* as of 15 October 1996) Former Yugoslav Republic of Macedonia (* as of 1 November 1997)  " _____ "	<b>11. Extension des effets du brevet européen</b> La présente demande est également réputée demande d'extension à tous les Etats non contractants de la CEE désignés dans la demande internationale, avec lesquels existaient, lors du dépôt de la demande, des "accords d'extension". Toutefois, l'extension ne produit ses effets que si la taxe d'extension prescrite est acquittée. Le demandeur se propose actuellement d'acquitter la taxe d'extension pour les Etats dont le nom est coché ci-dessous.  Slovénie (* à compter du 1 <sup>er</sup> mars 1994) Lituanie (* à compter du 5 juillet 1994) Lettonie (* à compter du 1 <sup>er</sup> mai 1995) Albanie (* à compter du 1 <sup>er</sup> février 1996) Roumanie (* à compter du 15 octobre 1996) Ex-République yougoslave de Macédoine (* à compter du 1 <sup>er</sup> novembre 1997)  " _____ "
I. Platz für Staaten, mit denen "Erstreckungsabkommen" nach Drucklegung dieser Formblatts in Kraft treten und die in der internationalen Anmeldung angegeben waren	I. Space for States with which "extension agreements" enter into force after this form has been printed and which were designated in the international application	II. Place pour des Etats à l'égard desquels des "accords d'extension" entreront en vigueur après l'impression du présent formulaire et qui ont été désignés dans la demande internationale
<input type="checkbox"/> <b>12. Automatischer Abbuchungsauftrag (Nur möglich für Inhaber von beim EPA geführten laufenden Konten)</b>  Das EPA wird beauftragt, nach Maßgabe der Vorschriften über das automatische Abbuchungsverfahren fällige Gebühren und Auslagen vom untenstehenden laufenden Konto abzubuchen.  Nummer des laufenden Kontos / Name des Kontoinhabers _____	<b>12. Automatic debit order (for EPO deposit account holders only)</b>  The EPO is hereby authorised, under the Arrangements for the automatic debiting procedure, to debit from the deposit account below any fees and costs falling due.  Deposit account number / Account holder's name _____	<b>12. Ordre de prélèvement automatique (uniquement possible pour les titulaires de comptes courants ouverts auprès de l'OEB)</b> Par la présente, il est demandé à l'OEB de prélever du compte courant ci-dessous les taxes et frais venant à échéance, conformément à la réglementation relative au prélèvement automatique.  N° du compte courant / Nom du titulaire du compte _____
<input type="checkbox"/> <b>13. Eventuelle Rückzahlungen auf das beim EPA geführte laufende Konto Nummer:</b>  _____ Name des Kontoinhabers _____	<b>13. Reimbursement, if any, to EPO deposit account number</b>  _____ Account holder's name _____	<b>13. Remboursements éventuels à effectuer sur la compte courant ouvert auprès de l'OEB numéro</b>  _____ Nom du titulaire du compte _____
<b>14. Unterschriften des (der) Anmelders (s) oder Vertreters</b>  Ort / Datum  Für Angestellte (Art. 133(3) EPÜ) mit allgemeiner Vollmacht: Nr. _____ (Bitte geben Sie den Namen der Angestellten bitte mit Angabe des Namens und des Dienstpostens an. Die Unterschriften der Angestellten müssen ebenfalls eingetragen werden.)	<b>14. Signature(s) of applicant(s) or representative</b>  Place / Date  For employees (Art. 133(3) EPC) having a general authorisation: No. <u>GA 43702</u> (Please type name(s) under signature(s). In the case of legal persons, the position of the signatories within the company should also be stated.)	<b>14. Signature(s) du (des) demandeur(s) ou du mandataire</b>  Lieu / Date  Pour les employés (art. 133(3) CEE) disposant d'un pouvoir général: N° _____ (Veuillez faire figurer le nom des employés sous la signature. Si l'un d'eux agit en tant que représentant d'une personne morale, la fonction de celui-ci doit également être indiquée.)

# **HUNTSMAN**

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Date August 14, 2001  
Direct Tel +44 (0) 121 420 5430  
Direct Fax +44 (0) 121 420 5437  
Your Ref  
Our Ref

**ASSIGNMENTS**

**REGISTERED POST**

European Patent Office  
Erhardtstrasse 27  
D-80298 Munich  
GERMANY

Dear Sirs

**ASSIGNMENT OF PATENT APPLICATIONS IN THE EPO & PCT  
FROM RHODIA CONSUMER SPECIALTIES LIMITED TO  
HUNTSMAN INTERNATIONAL LLC**

I enclose herewith a Certified Copy of the Patent Assignment containing the EPO and PCT Applications, which have been assigned, to Huntsman International LLC; these have the Country Codes of "WO" and "EP".

I also enclose a copy of the Power of Attorney authorising the undersigned representative to file these papers on behalf Huntsman International LLC.

If you require any further information or documentation please do not hesitate to let me know.

Yours faithfully  
For and on behalf of  
Huntsman International LLC

  
**R G M SAVIDGE**  
**AUTHORISED REPRESENTATIVE (GA 43702)**

Enc.



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Europäisches  
Patentamt

Eingangsstelle

European  
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Section

Office européen  
des brevets

Section de  
Dépôt

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GRANDE BRETAGNE



Records  
Book  
oldest new

Datum/Date

02/01/01

Zeichen/Ref./Réf.

MPD 315

Anmeldung Nr./Application No./Demande n°/Patent Nr./Patent No./Brevet n°

00945731.8 - -PCT/EP0005341

Anmelder/Applicant/Demandeur/Patentinhaber/Propriétaire/Titulaire

RHODIA CONSUMER SPECIALTIES LIMITED trading as ALB

### ENTRY INTO THE EUROPEAN PHASE BEFORE THE EUROPEAN PATENT OFFICE

NOTE: These notes describes the procedural steps required for entry into the European phase before the European Patent Office (EPO). You are advised to read them carefully; failure to take the necessary action in time can lead to your application being deemed withdrawn.

1. European patent application no. 00945731.8 has been allotted to the above-mentioned international patent application.
2. Applicants WITHOUT a residence or their principal place of of business within the territory of an EPC Contracting State may themselves initiate European processing of their international application, provided they do so before expiry of the 21st or 31st month from the the priority date (see also point 7 below).

ENTER  
EPO  
REGIONAL  
BY  
10 DECEMBER  
2001

During the European phase before the EPO as designated or elected Office, however, such applicants must be represented by a professional representative (Articles 133(2) and 134(7) EPC).

Procedural acts performed after expiry of the 21st or 31st month by a professional representative who acted during the international phase but is not authorised to act before the EPO have no legal effect and therefore lead to loss of rights.

Please note that a professional representative authorised to act before the EPO and who acted for the applicant during the international phase does not automatically become the representative for the European phase. Applicants therefore strongly advised to appoint in good time any representative they wish to initiate the European phase for them; otherwise, the EPO has to send all communications direct to the applicant.



3. Applicants WITH a residence or their principal place of business within the territory of an EPC Contracting State are not obliged to appoint a professional representative authorised to act before the EPO for the European phase before the EPO as a designated or elected Office.  
However, in view of the complexity of the procedure it is recommended that they do so.
4. Applicants and professional representatives are strongly advised to initiate the European phase using EPO Form 1200 (available free of charge from the EPO). This however is not compulsory.
5. TO ENTER THE EUROPEAN PHASE BEFORE THE EPO, the following acts must be performed. (NB: Failure validly to do so will entail loss of rights or other adverse legal consequences).
  - 5.1 If the EPO acting as DESIGNATED OFFICE under Article 22(1) PCT, applicants must, within 21 months from the date of filing or (where applicable) the earliest priority date:
    - a) Supply a translation of the international application into an EPO official language, if the International Bureau did not publish the application in such a language (Article 22(1) PCT and Rule 107(1)a) EPC).  
If the translation is not filed in due time, the international application is deemed to be withdrawn before the EPO (Article 24(1)(iii) PCT).
    - b) Pay the national basic fee and, where a supplementary European search report has to be drawn up, the search fee (Rule 107(1)c) and e) EPC).
    - c) Within six months from publication of the international search report, pay a designation fee for each designated Contracting State (Rule 107(1)d) EPC), and file a written request for examination and pay the examination fee (Rule 107(1)f) EPC).

Anmeldung Nr./Application No./Demande n°/Patent Nr./Polari No./Brevet n°	Blatt/Pages/Feuille
00945731.8	2



5.2 If the EPO is acting as ELECTED OFFICE under Article 39(1)a) PCT, applicants must, within 31 months from the date of filing or (where applicable) the earliest priority date:

- a) File a translation as per 5.1 a) above.
- b) Pay the fees as per 5.1 b) above.
- c) If the time limit under Article 79(2) EPC expires before the 31-month time limit, pay the designation fee for each designated Contracting State (Rule 107(1)d) EPC).
- d) If the time limit under Article 94(2) EPC expires before the 31-month time limit, file the written request for examination A N D pay the examination fee (Rule 107(1)f) EPC).
- e) Pay the renewal fee for the third year, if it falls due before the expiry of the 21-month time limit (Rule 107(1)g) EPC)

5.3 If the application documents on which the European grant procedure is to be based comprise more than ten claims, a claims fee is payable within the time limit under Rule 107(1) EPC for the eleventh and each subsequent claim (Rule 110(1) EPC). The fee can however still be paid within a period of grace of one month from notification of an EPO communication (Rule 110(2) EPC).

- 6. If the necessary fees are not paid in time, they may still be validly paid within a period of grace of one month from notification of an EPO communication, subject to payment at the same time of a surcharge for each late-paid fee (Rule 85a(1), 85b EPC). For the renewal fee, the period of grace is six months from the fee's due date (Article 86(2) EPC).
- 7. If the applicant had a representative during the application's international phase, the present notes will be sent to the representative, asking him to inform the applicant accordingly.

All subsequent communications will be sent to the applicant, or - if the EPO is informed of his appointment in time - to the applicants' European representative.

Anmeldung Nr./Application No./Demanda n°./Patent Nr./Patent No./Brevet n°.	Blatt/Page/feuille
00945731.8	3



8. For more details about time limits and procedural acts before the EPO as designated and elected Office, see the EPO brochure

How to get a European patent  
Guide for applicants - Part 2  
PCT procedure before the EPO - "EURO-PCT"

This brochure, the list of professional representatives before the EPO, Form 1200 and the latest fees are all on the internet under

<http://www.european-patent-office.org>.

RECEIVING SECTION



Anmeldung Nr./Application No./Demande n°/Patent Nr./Patent No./Brevet n°

00945731.8

Blatt/Seite/Feuille

4

**HUNTSMAN**

Huntsman Surface Sciences UK Limited

P O Box 9792  
210 - 222 Hagley Road West  
Oldbury  
West Midlands B68 0WA

Telephone +44 (0) 121 429 6700  
Facsimile +44 (0) 121 420 5700

**HUNTSMAN SURFACE SCIENCES UK LIMITED  
CHANGE OF DETAILS  
FROM 1 MARCH 2002**

Dear Sirs

You will be aware that our postal address recently changed to :

PO Box 9792  
210-222 Hagley Road West  
Oldbury  
West Midlands  
B68 0WA  
GREAT BRITAIN

If you have not yet implemented this change we would be grateful if you could update your records as soon as possible.

We would also inform you that with effect from 1 March 2002 our new telephone, and fax numbers will be as follows:

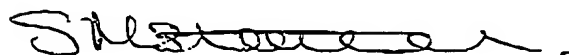
Main switchboard : +44 121 420 5812

Direct Lines : Mr R G M Savidge +44 121 420 5868  
Mrs S M Stevenson +44 121 420 5864  
+44 121 420 5802

Direct Fax : +44 121 420 5806

We would be grateful if you would use our direct lines whenever possible.

Yours faithfully  
For and on behalf of  
Huntsman Surface Sciences UK Limited



**MRS S M STEVENSON**  
**PATENTS & TRADEMARKS ADMINISTRATOR**

# Exhibit 5



# **HUNTSMAN**

Huntsman Surface Sciences UK Limited

P O Box 9761  
210 - 222 Hagley Road West  
Oldbury  
West Midlands B69 4XB

Telephone +44 (0) 121 429 6700  
Facsimile +44 (0) 121 420 5700

Date January 30, 2002  
Direct Tel +44 (0) 121 420 5431  
Direct Fax +44 (0) 121 420 5437  
Your Ref 2484658/mjc  
Our Ref MPD315/AU/  
RGMS/SMSL1

VIA FAX -  
CONFIRMATION BY POST

Davies Collison Cave  
1 Little Collins Street  
Melbourne, Victoria  
AUSTRALIA 3000

Dear Sirs

**AUSTRALIAN PATENT APPLICATION NO. 59716/00**  
**"PERSONAL CARE FORMULATIONS"**

I enclose the Report, as requested and confirm that no amendments have been made. The Applicant acquired its title from the first inventor by operation of UK law regarding the nature of his employment by the Applicant. I have asked the second Applicant to provide relevant details in respect of the second and third inventors.

Yours faithfully  
For and on behalf of  
Huntsman Surface Sciences UK Limited



**R G M SAVIDGE**  
**MANAGER, PATENTS & TRADEMARKS GROUP**

Enc.

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

by fax and post

PCT

NOTIFICATION OF TRANSMITTAL OF  
THE INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT

(PCT Rule 71.1)

To:

SAVIDGE, Roger, Gordon, Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury  
West Midlands B68 0NN  
GRANDE BRETAGNE

*File M 122.6*

Date of mailing  
(day/month/year)

10. 10. 01

Applicant's or agent's file reference

MPD315/PCT/RGMS

IMPORTANT NOTIFICATION

International application No.  
PCT/EP00/05341

International filing date (day/month/year)  
09/06/2000

Priority date (day/month/year)  
10/06/1999

Applicant

RHODIA CONSUMER SPECIALTIES LIMITED TRADING... et

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/



European Patent Office  
D-90293 Munich  
Tel. +49 89 2399 - 0 Tx: 523656 epmu d  
Fax: +49 89 2399 - 4465

Authorized officer

Hutlerer, G

Tel. +49 89 2399-6066





# PATENT COOPERATION TREATY

## PCT

### INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference <b>MPD315/PCT/RGMS</b>	<div style="display: flex; justify-content: space-between;"> <div><b>FOR FURTHER ACTION</b></div> <div>See Notification of Transmittal of International Preliminary Examination Report (Form PCT/PEA/410)</div> </div>	
International application No. <b>PCT/EP00/05341</b>	International filing date (day/month/year) <b>09/06/2000</b>	Priority date (day/month/year) <b>10/06/1999</b>
International Patent Classification (IPC) or national classification and IPC <b>A61K7/00</b>		
Applicant <b>RHODIA CONSUMER SPECIALTIES LIMITED TRADING... et</b>		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.15 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of _____ sheets.</p>		
<p>3. This report contains Indications relating to the following items:</p> <ul style="list-style-type: none"> <li>I <input checked="" type="checkbox"/> Basis of the report</li> <li>II <input type="checkbox"/> Priority</li> <li>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</li> <li>IV <input type="checkbox"/> Lack of unity of invention</li> <li>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</li> <li>VI <input type="checkbox"/> Certain documents cited</li> <li>VII <input type="checkbox"/> Certain defects in the international application</li> <li>VIII <input checked="" type="checkbox"/> Certain observations on the international application</li> </ul>		
Date of submission of the demand  <b>27/12/2000</b>	Date of completion of this report  <b>10.10.01</b>	
Name and mailing address of the International preliminary examining authority:   <b>European Patent Office</b> <b>D-80298 Munich</b> Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer  <b>Pregetter, M</b>  Telephone No. +49 89 2399 8719 <div style="text-align: right;">  </div>	

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/EP00/05341

**I. Basis of the report**

1. With regard to the elements of the international application (*Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report since they do not contain amendments (Rules 70.16 and 70.17)*):

Description, pages:

1-25 as originally filed

Claims, No.:

1-5 as originally filed

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).  
☐ the language of publication of the international application (under Rule 48.3(b)).  
☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.  
☐ filed together with the international application in computer readable form.  
☐ furnished subsequently to this Authority in written form.  
☐ furnished subsequently to this Authority in computer readable form.  
☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.  
☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:  
☐ the claims, Nos.:  
☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/EP00/05341

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes:	Claims
	No:	Claims 1-5
Inventive step (IS)	Yes:	Claims
	No:	Claims 1-5
Industrial applicability (IA)	Yes:	Claims 1-5
	No:	Claims

**2. Citations and explanations**

*see separate sheet*

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
*see separate sheet*

Re Item V

Reasoned statement under Article 35 (2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following documents:

D1: DE 14 67 825 A (CHESEBROUGH\_PONDS INC.) 2 January 1969 (1969-01-02)

- 2.1. The subject-matter of present claim 1 is not new according to Article 33(2) PCT. Document D1 already describes compositions comprising water, mineral oil, oil soluble surfactants and hydrophilic surfactants.

The compositions of D1 are described as transparent mineral oil-water-gels. The main use of these gels is as a "Frisiermittel".

The subject-matter of claim 1 cannot be clearly delimited from the compositions described in D1. See also the PCT-Guidelines III-4.8.

Example 1 discloses a composition comprising 20% mineral oil, 15% surfactant with an HLB value of between 12.7 and 15.0, 10% surfactant with an HLB value of between 1.6 and 7.6. The ratio of oil to oil soluble surfactant is 2:1, the ratio of oils soluble to hydrophilic surfactant is 1:1.5.

It is stated that 60°C lies just above the  $I_1/L_1$  transition temperature.

- 2.2. With regard to dependent claims 2-4 it is noted that a positive opinion can only be given, if dependent claims refer to independent claims that meet the requirements of the PCT.

3. The subject-matter of present claim 5 is not new according to Article 33(2) PCT. Document D1 describes a method for preparing compositions according to present claim 1 wherein mineral oil and the oil soluble surfactant are mixed and then a mixture of water and the hydrophilic surfactant is added. Water is added after the resulting mixture has been heated to a temperature above the "Gelpunkt" which is the  $I_1/L_1$  transition temperature. A cooling step is implicit. (p.10, 3rd paragraph - p.11, 2nd paragraph).

Re Item VIII

Certain observations on the international application

1. The subject-matter of present claim 1 is not clear (Article 6 PCT).
  - 1.1. The expression: "a proportion ..... based on the weight of ..." is not clear. Present claim 1 has been read as defining:
    - a) a proportion of from 8:1 to 1:5 of oil to oil soluble surfactant; and
    - b) a proportion of from 1:1 to 1:30 of oil soluble surfactant to hydrophilic surfactant.This interpretation of claim 1 is based on p.11, 3rd paragraph.
  - 1.2. The statement "adapted to form an  $I_1$  phase having  $I_1/L_1$  transition temperature greater than 25°C is not clear. Either the compounds comprised in the composition result in a composition with the defined effect, or an essential feature defining the claimed composition is missing.  
At present, all compositions comprising the defined compounds in the described proportions are considered to be relevant.

# Exhibit 6



**SMART & BIGGAR**

Intellectual Property &amp; Technology Law

FEB 25 2002

**To Fax No.:** 732-524-2808**Page 1 of:** 1**Attention:** Paula Hein**From:** Ron Ziola**Your file No.:** see below**Reply to Ottawa file no.:** 77414-103**Date:** February 25, 2002**Time:**P.O. Box 2999, Station D  
55 Metcalfe Street, Suite 900  
Ottawa, Canada K1P 5Y6

Tel.: (613) 232-2486

Fax: (613) 232-8440

Johnson & Johnson  
International Patent Law Division  
P.O. Box 1222  
New Brunswick, New Jersey 07903  
U.S.A.

Dear Madam:

Re: Proposed Entry of Canadian National Phase of  
PCT International Application  
Serial No. PCT /EP00/05341  
RHODIA CONSUMER SPECIALTIES LIMITED TRADING AS  
ALBRIGHT & WILSON SURFACTANTS EUROPE AND  
JOHNSON & JOHNSON CONSUMER COMPANIES, INC.  
Kevan Hatchman, et al.  
Your Ref: SPC0948

*Can - what should  
be done - drop  
corroto - you  
2/26/02*

Thank you for your facsimile dated February 19, 2002.

The status of this case is that we are awaiting your instructions to proceed as per Sujata Barot..

Yours very truly  
SMART & BIGGARRon Ziola  
Applications Supervisor

RZ:srm

**If there are any transmission problems, please call (613) 232-2486.**Original copy and any enclosures ☐ will be sent by ☐ mail  
☐ will not ☐ courier

The information contained in this transmission is confidential and only for the intended recipient identified above. If you are not the intended recipient, you are hereby notified that any dissemination or use of this communication is unlawful. If you have received this transmission in error, please immediately notify us by telephone (collect). Return the original message to us and retain no copy.

# Exhibit 7

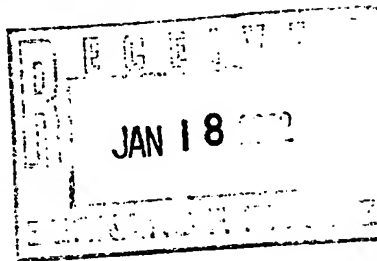
FRISHAUF, HOLTZ, GOODMAN, LANGER & CHICK, P.C.  
ATTORNEYS AT LAW

767 THIRD AVENUE, NEW YORK, N.Y. 10017-2023

LEONARD HOLTZ  
HERBERT GOODMAN  
THOMAS LANGER  
MARSHALL J. CHICK  
RICHARD S. BARTH  
DOUGLAS HOLTZ  
ROBERT P. MICHAL

OF COUNSEL:

STEPHEN H. FRISHAUF  
RICHARD M. GOLDBERG



*Ben was to file to Joe  
→ litigation*  
**CONFIRMATION**

*let file a  
CIP → TBP-529.*

TELEPHONE: (212) 319-4900

FACSIMILES:  
GROUP 3: (212) 319-5101  
GROUP 4: (212) 644-4834

WILLIAM R. WOODWARD  
(1914-1994)

January 15, 2002

E-MAIL: MJCHICK@FHGLC.COM

**VIA FACSIMILE - 1-732-524-5824**

Mr. Bernie Plantz  
JOHNSON & JOHNSON CONSUMER COMPANIES INC.  
One Johnson & Johnson Plaza  
New Brunswick, NJ 08933-7003

Re: National Phase of PCT/EP00/05341  
U.S. Serial No. 10/018,238  
Kevan HATCHMAN/HUNTSMAN INTERNATIONAL LLC and  
JOHNSON & JOHNSON CONSUMER COMPANIES INC.  
Huntsman Ref.: MPD315/US/RGMS/SMSL4  
Our Ref. : 01795/HG

Dear Mr. Plantz:

I have left a number of telephone messages for you and other people at Johnson & Johnson; however, to date, I have no information for use in this matter. Apparently Huntsman International LLC and Johnson & Johnson Consumer Companies Inc. are to be the assignees of the subject application in the United States. The application has already been filed and has been granted a Serial No. 10/018,238. The next step requires a post-filed Declaration and Power of Attorney. Inventors from J&J are apparently Elvin LUKENBACH, Laura MCCULLOCH and Benjamin WIEGAND; at least these are the three inventors with United States residences. In addition, it is usual to send an assignment for signature at the same time. Finally, there is an issue as to where the invention was made. If it was made outside of the United States then there is no problem. If any part of the invention was made within the United States the facts should be considered and, if necessary, action taken so that a valid U.S. patent can issue.

January 15, 2002

We were originally referred to Erin Harriman, but she is apparently on medical leave. Her assistant Beth was able to provide me with some current addresses for the U.S. inventors.

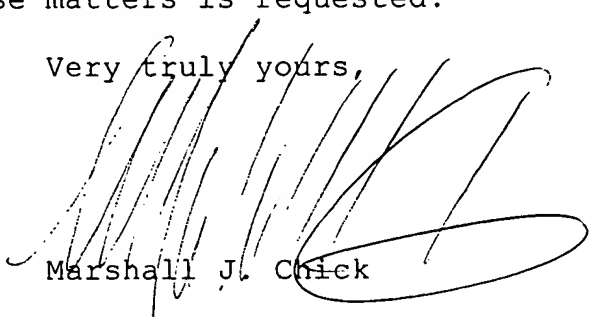
1. Concerning where the invention was made, I had received a call from a Jim Tracey who had indicated that he would get back to me in December. I have not heard anything further from him and my attempts to call him were unsuccessful. The only listing was apparently at a company called Vistacon and when I called that number I was directed to another person (I did not leave a telephone message). It is a fairly serious matter to file for a patent outside the United States when an invention is made, even in part, within the United States unless an export license is obtained. If reasonably prompt action is taken and there is a reasonable explanation, a retroactive license can be obtained. Otherwise a patent cannot issue in the United States.

2. With respect to the Assignment, the only address that I have for Johnson & Johnson Consumer Companies Inc. is the same address that I have for Huntsman International LLC. I would like to confirm that address or obtain a correct address so that I can complete the Assignment document.

3. Finally, is there a contact person to whom I can send the Declaration and Assignment for execution by the U.S. inventors.

Your assistance in these matters is requested.

Very truly yours,



Marshall J. Chick

MJC/ld

cc: Mr. Roger G.M. Savidge

# Exhibit 8

FRISHAUF, HOLTZ, GOODMAN, LANGER & CHICK, P.C.  
ATTORNEYS AT LAW

767 THIRD AVENUE, NEW YORK, N.Y. 10017-2023

RECEIVED

FEB 07 2002

Woodcock Washburn Kurtz  
Mackiewicz & Norris LLP

LEONARD HOLTZ  
HERBERT GOODMAN  
THOMAS LANGER  
MARSHALL J. CHICK  
RICHARD S. BARTH  
DOUGLAS HOLTZ  
ROBERT P. MICHAL

TELEPHONE: (212) 319-4900

FACSIMILES:  
GROUP 3: (212) 319-5101  
GROUP 4: (212) 644-4834

WILLIAM R. WOODWARD  
(1914-1994)

OF COUNSEL:  
STEPHEN H. FRISHAUF  
RICHARD M. GOLDBERG

VIA EXPRESS MAIL

E-MAIL: MJCHICK@FHGLC.COM

February 6, 2002

Wendy A. Choi, Esq.  
Woodcock Washburn  
One Liberty Place, 46<sup>th</sup> Floor  
Philadelphia, PA 19103

Re: U.S. Serial No. 10/018,238  
Kevan HATCHMAN, et al./HUNTSMAN INTERNATIONAL LLC and  
JOHNSON & JOHNSON CONSUMER  
COMPANIES INC.

Your Ref. : SPC-948 US  
J&J Ref. : JJ-0107  
Huntsman's Ref.: MPD315/US/RGMS/SMSL4  
Our Ref. : 01795/HG

Dear Wendy:

Referring to our telephone discussion earlier today, I am enclosing a copy of the subject application and of some citations which were sent to us by Roger Savidge.

There are no outstanding deadlines at this time (however see the numbered paragraphs below).

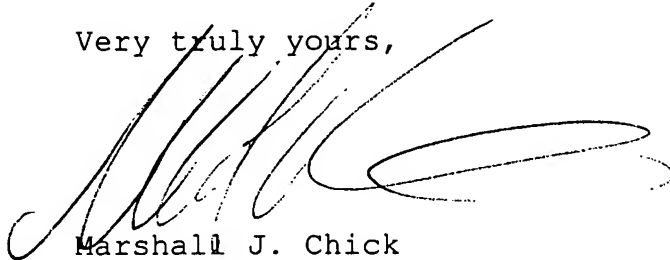
1. As I explained during our earlier telephone discussions, it appears that a retroactive foreign filing license must be obtained if a valid U.S. patent is to issue in this case. You had indicated that you were going to obtain a time line as to when the inventions were made and other facts. I assume that the responsibility for pursuing the retroactive license has been transferred to you along with the responsibility for the application. Petitioning the Patent Office for retroactive license under 37 CFR 5.25 should be done as quickly as possible after you are able to confirm that one is required.

2. No inventor declarations have been filed. there should be a requirement for such a declaration with a two month term received shortly. We will forward the requirement to you as soon as it is received. We assume that the power of attorney and address for such documents will be to you.

3. No assignment documents were filed. (We usually send them with the declaration to be signed at the same time).

4. Should you want to prepare declaration and assignment documents before the official requirement is made, please note that we confirmed the serial number with a telephone call to the Patent Office.

Very truly yours,

A handwritten signature in black ink, appearing to read "Marshall J. Chick", with a large, sweeping flourish extending to the right.

MJC/ld  
Encs.

J097 Rec'd PCT/PTO 07 DEC 2001

10/018238

RECEIPT ACKNOWLEDGED:

PCT National Phase Appln based on PCT/EP00/05341

Transmittal Letter Form PTO-1390 (in duplicate);  
Check No. 84229 for \$890.00; PRELIMINARY  
AMENDMENT; INFORMATION DISCLOSURE STATEMENT,  
includ. FORM PTO-1449A; also enclosed: Copy of  
WO 00/76460 A2; Int'l. Search Report  
(PCT/ISA/210); PCT/IB/308; PCT/IB/304; REQUEST  
FOR PUBLICATION OF ASSIGNMENT INFORMATION;  
CHANGE OF CORRESPONDENCE ADDRESS APPLN.  
Due Date: December 10, 2001 - MJC/ld

01795/HG - HATCHMAN et al - "PERSONAL CARE  
FORMULATIONS"

Express Mail Label No. EL 874 117 723 US  
Date of Deposit: December 7, 2001



TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

01795/HG

U.S. APPLICATION NO. (if known, see 37 CFR 1.3)

INTERNATIONAL APPLICATION NO.

INTERNATIONAL FILING DATE

PRIORITY DATE CLAIMED

PCT/EP00/05341

JUNE 9, 2000

JUNE 10, 1999

TITLE OF INVENTION

PERSONAL CARE FORMULATIONS

APPLICANT(S) FOR DO/EO/US Kevan HATCHMAN, Elvin LUKENBACH, Laura MCCULLOCH and Benjamin WIEGAND

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
  - a. ☒ is attached hereto (required only if not communicated by the International Bureau). (As WO 00/76460 A2)
  - b. ☒ has been communicated by the International Bureau.
  - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
  - a. ☐ is attached hereto.
  - b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☐ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
  - a. ☐ are attached hereto (required only if not communicated by the International Bureau).
  - b. ☐ have been communicated by the International Bureau.
  - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
  - d. ☐ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 20 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A **FIRST** preliminary amendment.
14. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
15. ☐ A substitute specification.
16. ☒ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.321 - 1.325.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

- (i) Copy of WO 00/76460 A2
- (ii) PCT/ISA/210 (Search Report)
- (iii) PCT/IB/304 (Priority Document Sent)
- (iv) PCT/IB/308 (Appln. sent to U.S.)
- (v) REQUEST FOR PUBLICATION OF ASSIGNMENT INFORMATION

Express Mail Mailing Label  
No.: EL 874 117 723 US  
Date of Deposit: December 7, 2001

I hereby certify that this paper is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR 1.10 on the date indicated above and is addressed to the Asst. Commissioner for Patents, Washington, D.C. 20231

*L. Davis*  
L. Davis

21. ☒ The following fees are submitted:**BASIC NATIONAL FEE** (37 CFR 1.492 (a) (1) - (5)):Neither international preliminary examination fee (37 CFR 1.482)  
nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO

and International Search Report not prepared by the EPO or JPO..... \$1040.00

International preliminary examination fee (37 CFR 1.482) not paid to  
USPTO but International Search Report prepared by the EPO or JPO ..... \$890.00International preliminary examination fee (37 CFR 1.482) not paid to USPTO  
but international search fee (37 CFR 1.445(a)(2)) paid to USPTO ..... \$740.00International preliminary examination fee (37 CFR 1.482) paid to USPTO  
but all claims did not satisfy provisions of PCT Article 33(1)-(4) ..... \$710.00International preliminary examination fee (37 CFR 1.482) paid to USPTO  
and all claims satisfied provisions of PCT Article 33(1)-(4) ..... \$100.00**ENTER APPROPRIATE BASIC FEE AMOUNT =**

\$ 890.00

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30  
months from the earliest claimed priority date (37 CFR 1.492(e)).

\$ --

**CLAIMS****NUMBER FILED****NUMBER EXTRA****RATE**

\$

Total claims

6 - 20 =

0

x \$18.00

\$ --

Independent claims

1 - 3 =

0

x \$84.00

\$ --

**MULTIPLE DEPENDENT CLAIM(S)** (if applicable)

+ \$280.00

\$

**TOTAL OF ABOVE CALCULATIONS =**

\$ 890.00

☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above  
are reduced by 1/2.

\$ --

**SUBTOTAL =**

\$ 890.00

Processing fee of \$130.00 for furnishing the English translation later than ☐ 20 ☐ 30  
months from the earliest claimed priority date (37 CFR 1.492(f)).

\$ --

**TOTAL NATIONAL FEE =**

\$ 890.00

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be  
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property +

\$ --

**TOTAL FEES ENCLOSED =**

\$ 890.00

Amount to be  
refunded:

\$

charged:

\$

a. ☒ A check in the amount of \$890.00 to cover the above fees is enclosed.b. ☐ Please charge my Deposit Account No. \_\_\_\_\_ in the amount of \$ \_\_\_\_\_ to cover the above fees.  
A duplicate copy of this sheet is enclosed.c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any  
overpayment to Deposit Account No. 06-1378. A duplicate copy of this sheet is enclosed.d. ☐ Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. Credit card  
information should not be included on this form. Provide credit card information and authorization on PTO-2038.**NOTE:** Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR  
1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

FRISHAUF, HOLTZ, GOODMAN LANGER & CHICK, P.C.  
767 Third Avenue - 25<sup>th</sup> Floor  
New York, NY 10017-2023

Tel. No. (212) 319-4900

Fax No. (212) 319-5101

Date: December 7, 2001



01933

PATENT TRADEMARK OFFICE

SIGNATURE

MARSHALL J. CHICK

NAME

26,853

REGISTRATION NUMBER

Attorney Docket No. 01795/HG

**IN THE UNITED STATES PATENT  
AND TRADEMARK OFFICE**

Applicant(s): Kevan HATCHMAN et al.

Serial No. : To be assigned (U.S.  
National Phase of  
PCT/EP00/05341  
filed June 9, 2000)

Filed : CONCOMITANTLY HEREWITH

For : PERSONAL CARE FORMULATIONS

Art Unit :

Examiner :

**ATTENTION BOX PCT**

**PRELIMINARY AMENDMENT FILED CONCOMITANT  
WITH NATIONAL PHASE PCT APPLICATION**

Assistant Commissioner for Patents  
Washington, D.C. 20231

S I R :

This is a PRELIMINARY AMENDMENT filed in the above-  
referenced national phase PCT application being filed  
concurrently herewith.


Please amend as follows:

**IN THE SPECIFICATION:**

Before the first paragraph of the specification insert the  
following paragraph: --This application is a U.S. National Phase  
Application under 35 USC 371 of International Application  
PCT/EP00/05341 (published in English) filed June 9, 1999.--

Express Mail Mailing Label  
No.: EL 874 117 723 US  
Date of Deposit: December 7, 2001

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date indicated above and is addressed to the  
Assistant Commissioner for Patents,  
Washington, D.C. 20231

  
Laraine Dobies

In the event that this Paper is late filed,  
and the necessary petition for extension of  
time is not filed concurrently herewith,  
please consider this as a Petition for the  
requisite extension of time, and to the  
extent not tendered by check attached  
hereto, authorization to charge the  
extension fee, or any other fee  
required in connection with this Paper  
to Account No. 06-1378.

IN THE CLAIMS:

Cancel claim 4.

Add the following new claims 6 and 7:

6. (New) A composition according to claim 1 wherein the oil comprises at least 16% based on the weight of oil, of a mineral oil.

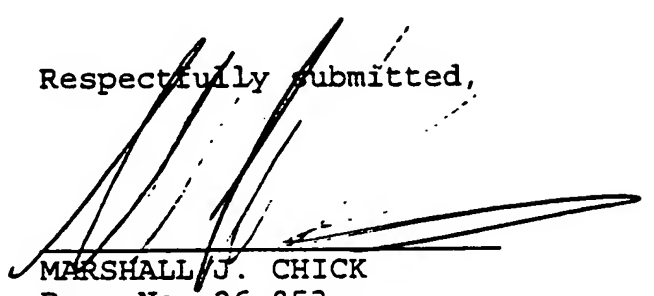
7. (New) A composition according to claim 3 wherein the oil comprises at least 16% based on the weight of oil, of a mineral oil.

R E M A R K S

Entry of this AMENDMENT and a favorable action on the merits are respectfully requested.

Frishauf, Holtz, Goodman,  
Langer & Chick, P.C.  
767 Third Ave., 25th Floor  
New York, NY 10017-2023  
Tel. No. (212) 319-4900  
Fax No.: (212) 319-5101  
MJC/ld

Respectfully submitted,



MARSHALL J. CHICK  
Reg. No. 26,853

Attorney Docket No. 01795/HG

**IN THE UNITED STATES PATENT  
AND TRADEMARK OFFICE**

Applicant(s): Kevan HATCHMAN et al.

Serial No. : To be assigned (U.S.  
National Phase of  
PCT/EP00/05341  
filed June 9, 2000)

Filed : CONCOMITANTLY HEREWITH

For : PERSONAL CARE FORMULATIONS

Art Unit :

Examiner :

**ATTENTION BOX PCT**

**INFORMATION DISCLOSURE STATEMENT  
FILED CONCOMITANT WITH NATIONAL  
PHASE PCT APPLICATION**

ASSISTANT COMMISSIONER FOR PATENTS  
Washington, D.C. 20231

S I R :

Submitted herewith are the following:


- (1) Copy of International Search Report issued in the  
above-referenced PCT application;
- (2) Copies of the cited publications are not enclosed  
herewith (this is a PCT national phase application);

and

- (3) Substitute Form PTO-1449A. It is requested that an  
initialed copy of the form PTO-1449 be returned to  
indicate that the publications listed therein have been  
considered and made of record.

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No.: EL 874 117 723 US  
Date of Deposit: December 7, 2001

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date indicated above and is addressed to the  
Assistant Commissioner for Patents,  
Washington, D.C. 20231

  
Laraine Dobies

In the event that this Paper is lat filed,  
and the necessary petition for extension of  
time is not filed concurrently herewith,  
please consider this as a Petition for the  
requisite extension of time, and to the  
extent not tendered by check attached  
hereto, authorization to charge the  
extension fee, or any other fee  
required in connection with this Paper  
to Account No. 06-1378.

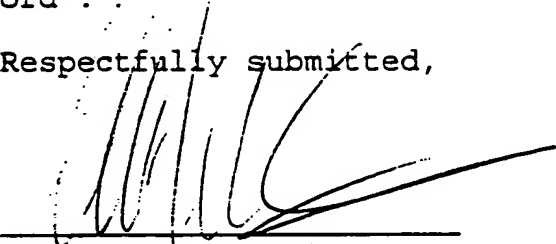
Each of the cited publications is considered relevant or material to the patentability of the present invention in view of the citation thereof in said communication and for the reasons stated in said communication. Said communication is in English.

This is being filed concurrently with the entry into the national phase and, therefore, is timely filed. No late fee is required.

It is therefore respectfully requested that the cited publications listed in the attached Substitute Form for PTO-1449A be considered and made "of record".

Frishauf, Holtz, Goodman,  
Langer & Chick, P.C.  
767 Third Ave., 25th Floor  
New York, NY 10017-2023  
Tel. No. (212) 319-4900  
Fax No.: (212) 319-5101  
MJC/ld

Respectfully submitted,

  
MARSHALL J. CHICK  
Reg. No. 26,853

Substitute for Form 1449A/PTO  <b>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</b>				Application Number		
				Filing Date		Herewith
				First Named Inventor		Kevan HATCHMAN et al.
				Group Art Unit		
				Examiner Name		
Sheet	1	of	2	Attorney Docket Number		01795/HG

## U.S. PATENT DOCUMENTS

Exam. Initials <sup>1</sup>	Cite No <sup>2</sup>	Document Number	Kind Code <sup>3</sup>	Name of Patentee or Applicant	Publication Date MM-DD-YYYY	Relevant Portion
		4,772,427 A		Dawson et al.	09-20-1988	
		5,756,108 A		Bruno et al.	05-26-1998	
		5,474,776 A		Hidenobu et al.	12-12-1995	

## FOREIGN PATENT DOCUMENTS

Exam. Initials <sup>1</sup>	Cite No <sup>2</sup>	Office <sup>4</sup>	Document Number <sup>4</sup>	Kind Code <sup>3</sup>	Name of Patentee or Applicant	Publication Date MM-DD-YYYY	Relevant Portion	T <sup>5</sup>
		DE	14 67 825 A			01-02-1969		
		EP	0 490 053 A			06-17-1992		

Examiner Signature		Date Considered	
-----------------------	--	--------------------	--

<sup>1</sup> EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 809. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

<sup>2</sup> Unique citation designation number. <sup>3</sup> See kinds of U.S. Patent Documents. <sup>4</sup> Enter Office that issued the document, by the two-letter code (WIPO Standard ST.3). <sup>5</sup> For Japanese patent documents, the indication of the year of the reign of the Emperor must precede the serial number of the patent document. <sup>6</sup> Kind of document by the appropriate symbols as indicated on the document under WIPO Standard ST. 16 if possible. <sup>7</sup> Place a check here if English translation is attached.

DATE MAILED: December 7, 2001



Substitute for Form 1449A/PTO  <b>INFORMATION DISCLOSURE STATEMENT BY APPLICANT</b>				Application Number		
				Filing Date		CONCOMITANT HEREWITH
				First Named Inventor		Kevan HATCHMAN et al.
				Group Art Unit		
				Examiner Name		
Sheet	2	of	2	Attorney Docket Number		01795/HG
<b>OTHER PRIOR ART - NON-PATENT LITERATURE DOCUMENTS</b>						
Examiner Initials <sup>1</sup>	Cite No. <sup>1</sup>	Include name of author (in CAPITAL LETTERS), title of article, title of item, date, page(s), volume-issue number(s), publisher, city and/or country where published				T <sup>2</sup>
		PATENT ABSTRACTS OF JAPAN, vol. 1999, No. 06, March 31, 1999 of JP 08 143420 A, June 4, 1996  RODRIGUEZ, et al. "Cubic phase-based concentrated emulsions", J. COLLOID INTERFACE SCI. (2000), 223(2), pp. 197-204, XP 00979610				
Examiner Signature				Date Considered		

\* EXAMINER: Initial if reference considered, whether or not citation is in conformance with MPEP 609. Draw line through citation if not in conformance and not considered. Include copy of this form with next communication to applicant.

<sup>1</sup> Unique citation designation number. <sup>2</sup> Place a check here if English translation is attached.

DATE MAILED: December 7, 2001

Attorney Docket No. 01795/HG

**IN THE UNITED STATES PATENT  
AND TRADEMARK OFFICE**

Applicant(s): Kevan HATCHMAN et al.

Serial No. : To be assigned (U.S.  
National Phase of  
PCT/EP00/05341  
filed June 9, 2000)

Filed : CONCOMITANTLY HEREWITH

For : PERSONAL CARE FORMULATIONS

Art Unit :

Examiner :

**ATTENTION BOX PCT**

**REQUEST FOR PUBLICATION OF  
ASSIGNMENT INFORMATION**

ASSISTANT COMMISSIONER FOR PATENTS  
Washington, D.C. 20231

S I R :

It is requested that the following assignment information be  
published as part of the 18-month publication of the present  
application:

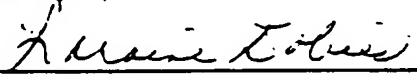
HUNTSMAN INTERNATIONAL LLC and  
JOHNSON & JOHNSON CONSUMER COMPANIES INC.

P.O. Box 9761  
210-222 Hagley Road West  
Oldbury  
West Midlands, B69 4XB  
ENGLAND

Frishauf, Holtz, Goodman,  
Langer & Chick, P.C.  
767 Third Ave., 25th Floor  
New York, NY 10017-2023  
Tel. No. (212) 319-4900  
Fax No.: (212) 319-5101  
MJC/ld

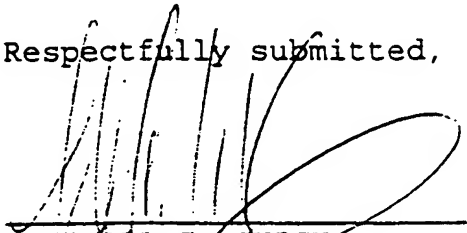
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Washington, D.C. 20231

  
Laraine Dooies

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time is not filed concurrently herewith,  
please consider this as a Petition for the  
requisite extension of time, and to the  
extent not tendered by check attached  
hereto, authorization to charge the  
extension fee, or any other fee  
required in connection with this Paper  
to Account No. 06-1378.

Respectfully submitted,

  
MARSHALL J. CHICK  
Reg. No. 26,853

Please type a plus sign (+) inside this box → ☐

PTO/SB/122 (10-2001)  
Approved for use through 10/31/2002. OMB 0631-0033  
U.S. Patent and Trademark Office, U.S. DEPARTMENT OF COMMERCE  
Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it displays a valid OMB control number.

## CHANGE OF CORRESPONDENCE ADDRESS Application

Address to:  
Assistant Commissioner for Patents  
Washington, D.C. 20231

Application Number	
Filing Date	Herewith
First Named Inventor	
Group Art Unit	
Examiner Name	
Attorney Docket Number	01795/HG

Please change the Correspondence Address for the above-identified application to:



Customer Number

01933

Type Customer Number here



01933

PATENT, TRADEMARK OFFICE

OR



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Individual Name

Address

Address

City

State

ZIP

Country

Telephone

Fax

This form cannot be used to change the data associated with a Customer Number. To change the data associated with an existing Customer Number use "Request for Customer Number Data Change" (PTO/SB/124).

I am the :



Applicant/Inventor.



Assignee of record of the entire interest.  
Statement under 37 CFR 3.73(b) is enclosed. (Form PTO/SB/96).



Attorney or Agent of record.



Registered practitioner named in the application transmittal letter in an application without an executed oath or declaration. See 37 CFR 1.33(a)(1). Registration Number 26,853

Typed or Printed  
Name

Marshall J. Chick, Reg. No. 26,853

Signature

Date

December 7, 2001

NOTE: Signatures of all the inventors or assignees of record of the entire interest or their representative(s) are required. Submit multiple forms if more than one signature is required. See below.



Total of \_\_\_\_\_ forms are submitted.

Burden Hour Statement: This form is estimated to take 3 minutes to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
21 December 2000 (21.12.2000)

PCT

(10) International Publication Number  
**WO 00/76460 A3**

(51) International Patent Classification: A61K 7/00.  
7/06, 7/50, 7/48, C11D 17/00, 1/94, 3/18

(74) Agent: SAVIDGE, Roger, Gordon, Madgwick; Rhodia  
Consumer Specialties Limited, 210-222 Hagley Road  
West, Oldbury, West Midlands B68 0NN (GB).

(21) International Application Number: PCT/EP00/05341

(22) International Filing Date: 9 June 2000 (09.06.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
9913408.2 10 June 1999 (10.06.1999) GB

(71) Applicant (for all designated States except US): RHO-  
DIA CONSUMER SPECIALTIES LIMITED trading  
as ALBRIGHT & WILSON SURFACTANTS EUROPE  
AND JOHNSON & JOHNSON CONSUMER COMPA-  
NIES INC [GB/GB]; 210-222 Hagley Road West, Oldbury,  
West Midlands B68 0NN (GB).

(81) Designated States (national): AE, AG, AL, AM, AT, AU,  
AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE,  
DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU,  
ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS,  
LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO,  
NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR,  
TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States (regional): ARIPO patent (GH, GM,  
KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian  
patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European  
patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,  
IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG,  
CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:

- With international search report.
- Before the expiration of the time limit for amending the  
claims and to be republished in the event of receipt of  
amendments.

(85) Date of publication of the international search report:  
25 May 2001

For two-letter codes and other abbreviations, refer to the "Guid-  
ance Notes on Codes and Abbreviations" appearing at the begin-  
ning of each regular issue of the PCT Gazette.

(72) Inventors; and

(75) Inventors/Applicants (for US only): HATCHMAN, Ke-  
van [GB/GB]; 5 Byland Close, Friarscroft, Bromsgrove,  
Worcestershire B61 7PL (GB). LUKENBACH, Elvin  
[US/US]; 160 Klinesville Road, Flemington, NJ 08822  
(US). MCCULLOCH, Laura [GB/US]; 18 Hampton  
Court 07920, Basking Ridge, NJ (US). WIEGAND,  
Benjamin [US/US]; 2028 Farmview Drive, Newton, PA  
18940 (US).

(54) Title: PERSONAL CARE FORMULATIONS

(57) Abstract: Personal care compositions contain at least 20 % water, 10 to 40 % total surfactant and 2 to 40 % of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (a) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (b) a hydrophilic surfactant having an HLB greater than 11 in a weight proportion of from 1:1 to 1:30 based on the weight of (a), said water surfactant and oil being present in proportions adapted to form an I<sub>1</sub> phase having an I<sub>1</sub>/L<sub>1</sub> transition temperature greater than 25 °C.

WO 00/76460 A3

# INTERNATIONAL SEARCH REPORT

Int l Application No

PCT/EP 00/05341

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/00 A61K7/06 A61K7/50 A61K7/48 C11D17/00  
C11D1/94 C11D3/18

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, CHEM ABS Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 14 67 825 A (CHESEBROUGH_PONDS INC.) 2 January 1969 (1969-01-02)	1-5
Y	page 1, paragraph 1 - paragraph 2 page 2, paragraph 3 page 3, paragraph 3 page 4, paragraph 4 -page 5, paragraph 1 page 9, paragraph 4 -page 11, paragraph 2 .. examples claims	1-5
A	US 4 772 427 A (DAWSON ANDREW F ET AL) 20 September 1988 (1988-09-20) abstract column 3, line 21 - line 24 column 3, line 41 -column 4, line 5 column 4, line 22 - line 34 examples 1,3,4,7 claims	1-3



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*S\* document member of the same patent family

Date of the actual completion of the international search

30 January 2001

Date of mailing of the international search report

14/03/2001

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl.  
Fax (+31-70) 340-3016

Authorized officer

Cielen, E

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

## Continuation of Box I.2

Present claims 1-5 relate to a composition and a method defined by reference to the parameter "an I1 phase having an I1/L1 transition temperature greater than 25 degrees C". The use of this parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameter the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search has been restricted to compositions having the properties of an I1 phase as described in the description (p. 1, par. 5, p. 5, par. 3 and 4, and p. 6, par. 2), namely compositions in the form of clear gels, ringed gels or having a viscous isotropic or a "VI" phase or a cubic liquid crystalline phase, or being immobile, non-Newtonian, optically isotropic and transparent.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

International Application No.

PCT/EP 00/05341

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>US 5 756 108 A (BIATRY BRUNO ET AL)  26 May 1998 (1998-05-26)  abstract  column 1, line 1 -column 2, line 16  column 2, line 63 -column 5, line 7  column 7, line 32 -column 8, line 4  examples 1,2,6  claims</p>	1-5
A	<p>US 5 474 776 A (KOYANAGI HIDENOBU ET AL)  12 December 1995 (1995-12-12)  abstract  column 2, line 4 - line 19  column 2, line 35 -column 5, line 30  table 3  examples 3,4  claims 1,2</p>	1-3
A	<p>PATENT ABSTRACTS OF JAPAN  vol. 1999, no. 06,  31 March 1999 (1999-03-31)  &amp; JP 03 143420 A (PROCTER &amp; GAMBLE  CO:THE), 4 June 1996 (1996-06-04)  abstract</p>	
A	<p>EP 0 490 053 A (WELLA AG)  17 June 1992 (1992-06-17)  abstract  page 2, line 35 -page 3, line 56</p>	1-3
P,Y	<p>RODRIGUEZ, CARLOS ET AL: "Cubic phase  -based concentrated emulsions"  J. COLLOID INTERFACE SCI. (2000), 223(2),  197-204,  XP000979610  abstract  page 197, column 1, paragraph 2 -column 2,  paragraph 4  figure 1  page 198, column 1, paragraph 5  page 199, column 1, paragraph 2 -page 200,  column 1, paragraph 2  page 202, column 1, paragraph 1 -page 203,  column 1, paragraph 1</p>	1-5

# INTERNATIONAL SEARCH REPORT

Information on patent family members

Int .ional Application No

PCT/EP 00/05341

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
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			PT 88252 A, B	10-11-1989
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			ZA 8805794 A	25-04-1990
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EP 0490053	A	17-06-1992	DE 4039063 A	11-06-1992
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			JP 4266811 A	22-09-1992
			US 5298240 A	29-03-1994



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7/06, 7/50, 7/48, C11D 17/00, 1/94, 3/18

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(21) International Application Number: PCT/EP00/05341

(22) International Filing Date: 9 June 2000 (09.06.2000)

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LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO,  
NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR,  
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patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE,  
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West Midlands B68 0NN (GB).

Published:

- With international search report.
- Before the expiration of the time limit for amending the  
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amendments.

(72) Inventors; and

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18940 (US).

(85) Date of publication of the international search report:  
25 May 2001

*For two-letter codes and other abbreviations, refer to the "Guid-  
ance Notes on Codes and Abbreviations" appearing at the begin-  
ning of each regular issue of the PCT Gazette.*

(54) Title: PERSONAL CARE FORMULATIONS

(57) Abstract: Personal care compositions contain at least 20 % water, 10 to 40 % total surfactant and 2 to 40 % of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (a) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (b) a hydrophilic surfactant having an HLB greater than 11 in a weight proportion of from 1:1 to 1:30 based on the weight of (a), said water surfactant and oil being present in proportions adapted to form an I<sub>1</sub> phase having an I<sub>1</sub>/L<sub>1</sub> transition temperature greater than 25 °C.

WO 00/76460 A3

# INTERNATIONAL SEARCH REPORT

International Application No

PCT/EP 00/05341

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 A61K7/00 A61K7/06 A61K7/50 A61K7/48 C11D17/00  
C11D1/94 C11D3/18

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ, CHEM ABS Data

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	DE 14 67 825 A (CHESEBROUGH_PONDS INC.) 2 January 1969 (1969-01-02)	1-5
Y	page 1, paragraph 1 - paragraph 2 page 2, paragraph 3 page 3, paragraph 3 page 4, paragraph 4 -page 5, paragraph 1 page 9, paragraph 4 -page 11, paragraph 2 .. examples claims	1-5
A	US 4 772 427 A (DAWSON ANDREW F ET AL) 20 September 1988 (1988-09-20) abstract column 3, line 21 - line 24 column 3, line 41 -column 4, line 5 column 4, line 22 - line 34 examples 1,3,4,7 claims	1-3
-/--		

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \*3\* document member of the same patent family

Date of the actual completion of the international search

30 January 2001

Date of mailing of the international search report

14/03/2001

Name and mailing address of the ISA

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NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax (+31-70) 340-3016

Authorized officer

Cielen, E

## FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

## Continuation of Box I.2

Present claims 1-5 relate to a composition and a method defined by reference to the parameter "an I1 phase having an I1/L1 transition temperature greater than 25 degrees C". The use of this parameter in the present context is considered to lead to a lack of clarity within the meaning of Article 6 PCT. It is impossible to compare the parameter the applicant has chosen to employ with what is set out in the prior art. The lack of clarity is such as to render a meaningful complete search impossible. Consequently, the search has been restricted to compositions having the properties of an I1 phase as described in the description (p. 1, par. 5, p. 5, par. 3 and 4, and p. 6, par. 2), namely compositions in the form of clear gels, ringed gels or having a viscous isotropic or a "VI" phase or a cubic liquid crystalline phase, or being immobile, non-Newtonian, optically isotropic and transparent.

The applicant's attention is drawn to the fact that claims, or parts of claims, relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

# INTERNATIONAL SEARCH REPORT

Int. Patent Application No  
PCT/EP 00/05341

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 756 108 A (BIATRY BRUNO ET AL) 26 May 1998 (1998-05-26) abstract column 1, line 1 -column 2, line 16 column 2, line 63 -column 5, line 7 column 7, line 32 -column 8, line 4 examples 1,2,6 claims	1-5
A	US 5 474 776 A (KOYANAGI HIDENOBU ET AL) 12 December 1995 (1995-12-12) abstract column 2, line 4 - line 19 column 2, line 35 -column 5, line 30 table 3 examples 3,4 claims 1,2	1-3
A	PATENT ABSTRACTS OF JAPAN vol. 1999, no. 06, 31 March 1999 (1999-03-31) & JP 08 143420 A (PROCTER & GAMBLE CO:THE), 4 June 1996 (1996-06-04) abstract	
A	EP 0 490 053 A (WELLA AG) 17 June 1992 (1992-06-17) abstract page 2, line 35 -page 3, line 56	1-3
P,Y	RODRIGUEZ, CARLOS ET AL: "Cubic phase -based concentrated emulsions" J. COLLOID INTERFACE SCI. (2000), 223(2), 197-204, XP000979610 abstract page 197, column 1, paragraph 2 -column 2, paragraph 4 figure 1 page 198, column 1, paragraph 5 page 199, column 1, paragraph 2 -page 200, column 1, paragraph 2 page 202, column 1, paragraph 1 -page 203, column 1, paragraph 1	1-5

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 00/05341

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
DE 1467825	A	02-01-1969	IT 950501 B	20-06-1973
US 4772427	A	20-09-1988	AT 399654 B	26-06-1995
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			ES 2007997 A	01-07-1989
			FI 883634 A,B,	02-06-1989
			FR 2623816 A	02-06-1989
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			JP 4266811 A	22-09-1992
			US 5298240 A	29-03-1994

## PATENT COOPERATION TREATY

PCT

NOTIFICATION CONCERNING  
SUBMISSION OR TRANSMITTAL  
OF PRIORITY DOCUMENT

(PCT Administrative Instructions, Section 411)

From the INTERNATIONAL BUREAU

To:

SAVIDGE, Roger, Gordon, Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury  
West Midlands B68 0NN  
ROYAUME-UNI

Date of mailing (day/month/year) 18 October 2000 (18.10.00)	
Applicant's or agent's file reference MPD315/PCT/RGMS	IMPORTANT NOTIFICATION
International application No. PCT/EP00/05341	International filing date (day/month/year) 09 June 2000 (09.06.00)
International publication date (day/month/year) Not yet published	Priority date (day/month/year) 10 June 1999 (10.06.99)
Applicant RHODIA CONSUMER SPECIALTIES LIMITED et al	

- The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
- An asterisk (\*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

<u>Priority date</u>	<u>Priority application No.</u>	<u>Country or regional Office or PCT receiving Office</u>	<u>Date of receipt of priority document</u>
10 June 1999 (10.06.99)	9913408.2	GB	09 Augu 2000 (09.08.00)

The International Bureau of WIPO 34, chemin des Châmbettes 1211 Geneva 20, Switzerland	Authorized officer  Athina Nickitas-Etienne
Facsimile No. (41-22) 740.14.35	Telephone No. (41-22) 338.83.38

# PATENT COOPERATION TREATY

1. For copy to J. Zahra 3/1/2001  
From the INTERNATIONAL BUREAU

PCT  
2 File M. d. d. l.

## NOTICE INFORMING THE APPLICANT OF THE COMMUNICATION OF THE INTERNATIONAL APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

To:

SAVIDGE, Roger, Gordon, Madgwick  
Rhodia Consumer Specialties Limited  
210-222 Hagley Road West  
Oldbury  
West Midlands B68 0NN  
ROYAUME-UNI

Date of mailing (day/month/year)

21 December 2000 (21.12.00)

Applicant's or agent's file reference

MPD315/PCT/BGMS

### IMPORTANT NOTICE

International application No.

PCT/EP00/05341

International filing date (day/month/year)

09 June 2000 (09.06.00)

Priority date (day/month/year)

10 June 1999 (10.06.99)

Applicant

RHODIA CONSUMER SPECIALTIES LIMITED trading as ALBRIGHT & WILSON  
SURFACTANTS EUROPE AND JOHNSON & JO et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:  
AG, AU, DZ, KP, KR, MZ, US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE, AL, AM, AP, AT, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EA, EE, EP, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, OA, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on 21 December 2000 (21.12.00) under No. WO 00/76460

### REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

### REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

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1211 Geneva 20, Switzerland

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Facsimile No. (41-22) 740.14.35

Telephone No. (41-22) 338.83.38

1 Fax copy to - 2/1/00  
2 FBI MPD 315 ACT. Bo = 6



## PCT

(10) International Publication Number  
**WO 00/76460 A2**

**Benjamin [US/US]:** 2028 Farmview Drive, Newton, PA 18940 (US).

(74) Agent: SAVIDGE, Roger, Gordon, Madgwick; Rhodia Consumer Specialties Limited, 210-222 Hagley Road West, Oldbury, West Midlands B68 0NN (GB).

(31) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(30) Priority Data: 9913408.2 10 June 1999 (10.06.1999) GB

(8-4) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

(75) **Inventors/Applicants (for US only):** HATCHMAN, Kevin [GB/GB]; 5 Byland Close, Friarscroft, Bromsgrove, Worcestershire B61 7PL (GB). LUKENBACH, Elvin [US/US]; 160 Klinesville Road, Flemington, NJ 08822 (US). MCCULLOCH, Laura [GB/US]; 18 Hampton Court 07920, Basking Ridge, NJ (US). WIEGAND,

— Without international search report and to be republished upon receipt of that report.

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(57) Abstract: Personal care compositions contain at least 20 % water, 10 to 40 % total surfactant and 2 to 40 % of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (a) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (b) a hydrophilic surfactant having an HLB greater than 11 in a weight proportion of from 1:1 to 1:30 based on the weight of (a), said water surfactant and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25 °C.

**WO 00/76460 A2**



## PERSONAL CARE FORMULATIONS

The present invention relates to shampoo or cleaning compositions suitable for personal care applications in the form of  $I_1$  mesophase systems containing dispersed oil.

Dispersing oil in aqueous shampoo and body wash formulations has presented problems. To prevent the oil phase separating it must either be: (A) emulsified which involves dispersing the oil as colloidal single droplets; (B) microemulsified which involves forming a micellar solution with oil incorporated into surfactant micelles; (C) suspended in a structured surfactant system which typically comprises a dispersion of a surfactant mesophase in aqueous electrolyte; or (D) incorporated into a water soluble solid, pasty or gelatinous composition.

With the exception of microemulsions which are clear, thermodynamically stable, micellar solutions, the foregoing systems are necessarily opaque and contain the oil dispersed in a relatively coarse form, which does not deposit satisfactorily on skin or hair.

However microemulsions are difficult to formulate using the surfactants which are most effective in body wash and other personal care formulations and contain relatively low concentrations of surfactant.

We have now discovered that oil may be stably incorporated into the structure of an  $I_1$  phase to form a clear gel-like composition which contains higher concentrations of surfactant and oil than conventional microemulsions, but which dissolves in water to form a microemulsion. The novel oil-in- $I_1$  compositions also form microemulsions on heating.

Surfactants are known to form mesophases or liquid crystal phases at concentrations above approximately 30% by weight based on the weight of water and surfactant. Mesophases are phases which exhibit a degree of order intermediate between typical liquids and solids. Generally mesophases combine long range order associated with crystals, with fast molecular motion common to liquids.

The formation of detergent mesophases is well documented. Different surfactants and surfactant mixtures differ widely in their ability to form the numerous different mesophases, and in respect of the conditions of concentration and temperature at which they are formed. For a typical surfactant of the type normally used in cleaning products the following mesophases are usually observed. The concentrations given are illustrative only and may vary considerably from one surfactant or surfactant mixture to the next.

Below approximately 30% surfactant an isotropic  $L_1$  phase is formed (with micelles of surfactant in water). Above 30% surfactant many detergents form a M phase which is not normally used in personal care applications since it does not show suitable flow characteristics and is difficult to dissolve or disperse in water. Above the concentrations required to form an M phase, but usually at concentrations of less than 80% active surfactant, i.e. 60%-80% a G-phase is formed. At concentrations higher than those required to form a G-phase, i.e. typically greater than 80% active surfactant, most surfactants form a hydrated solid, and some, especially non-ionic surfactants form a liquid phase containing dispersed micelle sized droplets of water - an inverted micellar solution known as an  $L_2$  phase.  $L_2$  detergent systems do not disperse readily in water and have a tendency to form undesirable gels, e.g. M phases, on dilution.

Some surfactants form viscous isotropic or VI phases. These are immobile phases usually with a vitreous appearance, and have been relatively little studied compared to the other phases discussed above. They have been virtually ignored in the context of formulating cleaning compositions because most of the surfactants and surfactant systems which are commonly used in cleaning compositions do not form VI phases. at least at

normal temperatures, or form them only within narrow concentration ranges and because their known properties as immobile gels has deterred formulators from investigating them. They are recognised as being the most viscous of the lyotropic mesophases.

The different surfactant phases can be recognised by a combination of appearance, rheology, textures under the microscope, electron microscopy and x-ray diffraction or neutron scattering. A detailed description, with illustrations, of the difference textures observable using a polarising microscope, is to be found in the paper by Rosevear JAOCS Vol 31, p628.

The following terms may require explanation or definition:

The "hydrophilic: lipophilic balance", or "HLB" value is used as a measure of the relative affinities of the surfactants for water and oil respectively and correlates with their effectiveness as emulsifiers. HLB value can easily be calculated for alcohol ethoxylates since it is one fifth of the weight percent of ethylene oxide based on the total mole weight. Other surfactants can be assigned equivalent values by applying more complicated formulae or by measuring their relative affinity for water and oil. An HLB value of 20 represents a completely water soluble oil insoluble surfactant, while an HLB value of 0 represents a completely oil soluble and water insoluble surfactant.

"Optically isotropic" surfactant phases do not normally tend to rotate the plane of polarisation of plane polarised light. If a drop of sample is placed between two sheets of optically plane polarising material whose planes are at right angles, and light is shone on to one sheet, optically isotropic surfactant samples do not appear substantially brighter than their surrounding when viewed through the other sheet. Optically anisotropic materials appear substantially brighter. Optically anisotropic mesophases typically show characteristic textures when viewed through a microscope between crossed polarisers, whereas optically isotropic phases usually show a featureless continuum.

"Newtonian liquids" have a viscosity which remains constant at different shear rates. For the purpose of this specification, liquids are considered Newtonian if the viscosity does not vary substantially at shear rates up to  $1000 \text{ sec}^{-1}$ .

"Lamellar" phases are phases which comprise a plurality of bilayers of surfactant arranged in parallel and separated by liquid medium. They include both solid phases and the typical form of the liquid crystal G-phase. G-phases are typically pourable, non-Newtonian, anisotropic products. They are typically viscous-looking, opalescent materials with a characteristic "smeary" appearance on flowing. They form characteristic texture under the polarising microscope and freeze fractured samples have a lamellar appearance under the electron microscope. X-ray diffraction or neutron scattering similarly reveal a lamellar structure, with a principal peak typically between 4 and 10nm, usually 5 to 6nm. Higher order peaks, when present occur at double or higher integral multiples of the Q value of the principal peak. Q is the momentum transfer vector and is related, in the case of lamellar phases, to the repeat spacing d by the equation  $Q = \frac{2n}{d} [\pi]$  where n is the order of the peak.

G-phases, however, can exist in several different forms, including domains of parallel sheets which constitute the bulk of the typical G-phases described above and spherulites formed from a number of concentric spheroidal shells, each of which is a bilayer of surfactant. In this specification the term "lamellar" will be reserved for compositions which are at least partly of the former type. Opaque compositions at least predominantly of the latter type in which the continuous phase is a substantially isotropic solution containing dispersed spherulites are referred to herein as "G-phase compositions". G-phases are sometimes referred to in the literature as  $L_{(\alpha)}$  phases.

$L_1$ -phases are mobile, optically isotropic, and typically Newtonian liquids which show no texture under the polarising microscope. Electron microscopy is capable of resolving the texture of such phases only at very high magnifications, and X-ray or neutron scattering normally gives only a single broad peak typical of a liquid structure, at very small angles

close to the reference beam. The viscosity of an  $L_1$ -phase is usually low, but may rise significantly as the concentration approaches the upper phase boundary.

"M-phases" are typically immobile, anisotropic products resembling low melting point waxes. They give characteristic textures under the polarising microscope, and a hexagonal diffraction pattern by X-ray or neutron diffraction which comprises a major peak, usually at values corresponding to a repeat spacing between 4 and 10nm, and sometimes higher order peaks, the first at a Q-value which is  $3^{0.5}$  times the Q-value of the principal peak and the next double the Q-value of the principal peak. M-phases are sometimes referred to in the literature as H-phases.

The viscous isotropic or "VI" phases are typically immobile, non-Newtonian, optically isotropic and are typically transparent, at least when pure. VI phases have a cubic symmetrical diffraction pattern, under X-ray diffraction or neutron scattering, with a principal peak and higher order peaks at  $2^{0.5}$  and  $3^{0.5}$  times the Q-value of the principal peak.

These cubic liquid crystalline phases are sometimes observed immediately following the micellar phase at ambient temperature as the concentration of surfactant is increased. It has been proposed that such VI phases, sometimes referred to as  $I_1$  phase, may arise from the packing of micelles (probably spherical) in a cubic lattice. At ambient temperature a further increase in surfactant concentration usually results in hexagonal phase ( $M_1$ ), which may be followed by a lamellar phase (G).  $I_1$  phases, when they occur, are usually only observed over a narrow range of concentrations, typically just above those at which the  $L_1$ -phase is formed. The location of such VI phases in a phase diagram suggests that the phase is built up of small closed surfactant aggregates in a water continuum.

An inverse form of the  $I_1$  phase (the  $I_2$  phase) has also been reported, possibly between the inverse hexagonal ( $M_2$ ) and  $L_2$  phases. It consists of a surfactant continuum containing a cubic array of inverted micelles. An alternative form of the VI phase called

the  $V_1$  phase has been observed at concentrations between the M and G phases and may comprise a bicontinuous system. This may exhibit an even higher viscosity than the  $I_1$ . An inverse phase, the  $V_2$  phase, between the G and  $M_2$  phases has also been postulated.

VI phases are typically examples of "ringing gels". When a jar or beaker containing such a phase is sharply struck, a distinctive vibration can be felt in the composition.

The  $I_1/L_1$  transition temperature will be referred to herein as the melting point of the  $I_1$  phase for convenience, although it is not strictly speaking the melting point since the VI phases are not solids.

All references herein to the formation or existence of specific phases or structures are to be construed, unless the context requires otherwise, as references to their formation or existence at 20°C.

Hexagonal gels (M-phase) have been referred to in the prior art as cleaning compositions, e.g. GB 2 179 055, EP I 153 837 and colloidal gels formed with gelling agents such as synthetic polymers or gelatin have also been suggested, e.g. US 4 465 663.

However these compositions cannot be readily dissolved in water to form microemulsions. They are moreover usually opaque and of an unattractive appearance and often require the presence of solvents such as glycols which add to the cost and are environmentally undesirable.

The use of a type of ringing gel to suspend oil for cosmetic or pharmaceutical applications was described in US 4 026 818 but the formulation requires the presence of hydroxylic solvents and utilises a surfactant system which is unsuitable for shampoo applications. EP O 598 335 describes the use of various cubic phases including  $I_1$  phases as laundry prespotters and for other cleaning formulations. It does not suggest how such phases could be used to suspend oil or form microemulsions. Normally attempts to

suspend oil in surfactant mesophases result in coarse droplets of oil being suspended in the aqueous phase of a structured surfactant.

Our invention provides a concentrated personal cleansing composition comprising, by weight of the composition, at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil, such as a mineral, fatty ester, glyceride, terpene or silicone oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.

The surfactants are preferably selected to provide an  $I_1$  phase over a comparatively broad surfactant concentration range e.g. more than  $\pm 5\%$  or greater, which range typically lies above 15% by weight total surfactant based on the weight of the composition e.g. between 20% and 40% by weight surfactant usually between 25% and 60%.

The surfactants are preferably selected to provide an  $I_1$  phase which melts above 30°C e.g. above 35°C, most preferably above 40°C. Preferably the  $I_1$  phase melts at a temperature substantially below 100°C, e.g. below 90°C, more preferably below 80°C, most preferably below 70°C, especially below 60°C, typically below 55°C, usually below 50°C.

The surfactant mixture preferably has a mean HLB based on the molar proportions of the components between 10 and 15 e.g. 11 to 14. The surfactants preferably comprise non-ionic surfactants such as ethoxylated alcohols. It has been found that highly ethoxylated fatty alcohols, e.g. more than 10 EO groups, preferably more than 15 EO groups, especially 18 to 50 EO groups form  $I_1$  phases particularly readily.

Other non-ionic surfactants which may be present include:-

alkyl phenol ethoxylates, fatty acid ethoxylates, fatty acid monoalkylolamide ethoxylates, fatty alcohol propoxylates, fatty anime alkoxyates and fatty acid glyceryl ester ethoxylates. Other non-ionic compounds suitable for inclusion in compositions of the present invention include mixed ethylene oxide propylene oxide block copolymers, low relative molecular mass polyethylene glycols e.g. PEG600 and PEG200, ethylene glycol monoesters, amine oxides and alkyl polyglycosides, alkyl sugar esters including alkyl sucrose esters and alkyl oligosaccharide ester, alkyl capped polyvinyl alcohol and alkyl capped polyvinyl pyrrolidone.

Compositions of the invention may also comprise anionic surfactants, in addition to or instead of non-ionic surfactants. Anionic surfactant may comprise a  $C_{10-20}$  alkyl benzene sulphonate or an alkyl ether sulphate which is preferably the product obtained by ethoxylating a natural fatty or synthetic  $C_{10-20}$  e.g. a  $C_{12-14}$  alcohol with from 1 to 20, preferably 2 to 10 e.g. 3 to 4 ethyleneoxy groups, optionally stripping any unreacted alcohol, reacting the ethoxylated product with a sulphating agent and neutralising the resulting alkyl ether sulphuric acid with a base. The term also includes alkyl glyceryl sulphates, and random or block copolymerised alkyl ethoxy/propoxy sulphates.

The anionic surfactant may also comprise, for example,  $C_{10-20}$  e.g.  $C_{12-18}$  alkyl sulphate.

The surfactant may comprise a  $C_{8-20}$  e.g.  $C_{10-20}$  aliphatic soap. The soap may be saturated or unsaturated, straight or branched chain.

Preferred examples include dodecanoates, myristates, stearates, oleates, linoleates, linoleates and palmitates and coconut and tallow soaps.

The surfactant may include other anionic surfactants, such as olefin sulphonates, paraffin sulphonates, taurides, isethionates, ether sulphonates, ether carboxylates, aliphatic ester sulphonates e.g. alkyl glyceryl sulphonates, sulphosuccinates or sulphosuccinamates.



The cation of any anionic surfactant is typically sodium but may alternatively be potassium, lithium, calcium, magnesium, ammonium, or an alkyl ammonium having up to 6 aliphatic carbon atoms including isopropyl ammonium, monoethanol ammonium, diethanol ammonium, and triethanol ammonium.

Ammonium and ethanol ammonium salts are generally more soluble than the sodium salts. Mixtures of the above cations may be used.

The composition may contain amphoteric surfactants such as betaines sulphobetaines, amido betaines or imidazoline betaines.

The  $I_1$  phase may be conveniently prepared by mixing the oil and oil soluble surfactant and adding sufficient water to the water soluble surfactant to maintain a lamellar phase. The oil and oil soluble surfactant may be stirred into the lamellar composition at elevated temperature, above the melting point of the desired  $I_1$  phase. The composition is then diluted with hot water until a microemulsion is formed and then cooled to solidify it into the  $I_1$  phase.

The oil is preferably a mineral oil (e.g. a low molecular weight petroleum ether having, for example, a boiling point below 120°C e.g. below 100°C especially below 80°C) or a lower molecular weight fatty ester (e.g. one having less than 25 carbon atoms) such as isopropyl esters of lauric isostearic or palmitic acids or their ethyl analogues. Other oils, including higher mol weight fatty esters, e.g. oleyl oleate, fatty glycerides, terpene oils such as limonene or silicone oils may present difficulties in providing clear compositions. Such oils can nevertheless be incorporated in clear formulations by blending with sufficient mineral oil (preferably low molecular weight mineral oil). The amount required varies according to the nature of the oil. Typically the blend contains at least 16%, based on the total weight of oil, of the mineral oil, especially 30 to 80%, typically 40 to 60%. Particularly preferred are vegetable oils such as coconut, evening primrose, groundnut, meadow foam, apricot kernel, peach kernel, avocado, jojoba and olive oil.

Oil soluble cosmetic or topical pharmaceutical ingredients may be dissolved in the oil including antiseptics, styptics, antidandruff agents such as zinc omadine (zinc pyrithione) and selenium disulphide, proteins, emollients such as lanolin, isopropyl myristate, glyceryl isostearate or propylene glycol distearate, dyes, perfumes and waxes. Water insoluble particulate solids including exfoliants such as talc, clays, polymer beads, sawdust, silica, seeds, ground nutshells and dicalcium phosphate, pearlisers such as mica or glycerol or ethylene glycol mono- or di-stearate, glitter additives and sunscreens such as titanium dioxide may be dispersed in the hot microemulsion prior to cooling. Porous particles (so called micro-sponges) containing absorbed active ingredients or gelatin or other microcapsules may be suspended. Other active ingredients which may be suspended include insect repellants and topical pharmaceutical preparations, e.g. preparations for treatment of acne, fungicides for athlete's foot or ringworm or antiseptics or antihistamines. Pigments, such as the iron oxides, may also be added.

Electrolytes tend to break  $I_1$  phase structure and are preferably present in concentrations below 10% based on total weight of the compositions, more preferably below 5%, e.g. 0 to 3%, most preferably 0 to 1%. Generally we prefer that electrolyte be substantially absent. Adventitious chloride or sulphate present as impurities in the surfactant can be tolerated. Small amounts of builder such as citrates, pyrophosphates, polyphosphates may optionally be included.

Water soluble solvents are generally undesirable and are not required to form stable  $I_1$  structures according to the invention. We therefore prefer that they should be substantially absent. Although small amounts of, for example, ethanol or propanol or of a water miscible polyhydric alcohol or alcohol ester may sometimes be desired for special purposes, they are preferably present in amounts less than 5% by weight, more preferably less than 3% by weight, most preferably less than 2% by weight, e.g. less than 1% by weight.

The composition may optionally contain hydrotropes such as sodium lower alkyl benzene sulphonate e.g. sodium toluene, xylene or cumene sulphonate or urea, however these are not generally necessary and are not generally preferred. We prefer that these should be present in quantities less than 5% by weight, more preferably less than 4%, especially less than 2% e.g. 0 to 1%. They may be useful occasionally to avoid haziness of the gel.

The total amount of water is preferably from 25 to 60% by weight of the composition, more preferably 30 to 50%, e.g. 35 to 50%. The total weight percentage of surfactant based on the weight of the composition is preferably from 15 to 35%, e.g. 20 to 30%. The proportion of oil is preferably greater than 5%, more preferably greater than 8%, e.g. 10 to 30%, especially 15 to 25% by weight based on the weight of the composition. The oil soluble surfactant is preferably present in a proportion of more than 1:5 based on the weight of oil, more preferably from 1:2 to 5:1. The oil soluble surfactant preferably has an HLB of from 3 to 9 e.g. 4 to 8.

The weight ratio of water soluble surfactant to oil soluble surfactant is preferably 1:1 to 30:1, more preferably 2:1 to 20:1, typically 3:1 to 15:1, e.g. 4:1 to 10:1. The water soluble surfactant preferably has an HLB greater than 12, more preferably greater than 13, especially 14 to 19.

The product may be cast into shaped bodies or formed into particles or granules, e.g. by spray cooling a hot solution of the  $L_1$  phase formed on melting the composition.

The composition may be converted into a microemulsion phase by addition of water, by heating above the melting point or by adding electrolyte such as salt and the invention includes  $L_1$  phases when so prepared.

The invention will be illustrated by the following examples:

**Example 1**

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	20	20
"EMPICOL"® 0251/70J (70%)	11.2	16
"EMPIGEN"® BB (30%)	4.8	16
"GLUCAPON"® 215 CS UP (65%)	6	9.2
"EMPILAN"® KB2 (100%)	7.5	7.5
SODIUM CHLORIDE (100%)	2	2
PERFUME (100%)	0.5	0.5
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.1	0.1
CITRIC ACID (100%)	0.2	0.2
BENZOIC ACID (100%)	0.3	0.3
SODIUM HYDROXIDE (47%)	0.1	0.2
WATER	---	Balance

The method of mixing comprised the following steps:-

1. Charge 50% of water
2. Heat to 60°C
3. Add EDTA, sodium benzoate, citric acid and 47% NaOH dissolve with stirring
4. Add "EMPIGEN" BB
5. Add mineral oil and disperse with stirring
6. Add "EMPILAN" KB 2 and mix thoroughly
7. Add "EMPICOL" 0251/70j
8. Add remaining water
9. Add "GLUCAPON" 215 CS UP
10. Add further KB 2 until clear
11. Cool
12. Add evaporated water
13. Adjust pH

#### Physical Data

pH (10%)	: 5.5 ± 0.1	Density @ 20°C	: 1.0 ± 0.1 g cm <sup>-3</sup>
Solids (%)	: ~ 53% (typical)	Appearance	: Clear or Hazy Gel
Odour	: Characteristic	Set Point (typical)	: 30°C
Viscosity @ 20°C : N/A			

The product was examined by x-ray diffraction and exhibited peaks at 13.145nm (intense and sharp), 7.943nm (ill defined) and 6.355nm (small), indicating cubic symmetry, and formed a clear microemulsion on dilution or heating. The latter gave good even distribution of oil applied to skin.

#### Example 2

The following ingredients were mixed at 60°C and cooled to form a ringing gel:

<u>Component</u>	<u>Solids (%)</u>	<u>w/w (%)</u>
MINERAL OIL (100%)	15	15
"EMPICOL"® CDL30J/35 (22%)	8	35.4
"EMPIGEN"® BB (30%)	8	26.7
"EMPICOL"® 0785 (40%)	2	5
"EMPILAN"® KB2 (100%)	6	6
"EMPILAN"® KB6 (100%)	6	6
CITRIC ACID (100%)	0.5	0.5
PERFUME (100%)	0.2	0.2
ETHYLENE DIAMINE TETRACETIC ACID (100%)	0.2	0.2
"KATHON"®	---	0.2
WATER	---	Balance
<b>TOTAL</b>	<b>45.8</b>	<b>100</b>

#### Physical Data

Appearance	: Clear Liquid/Gel	Odour	: Characteristic Odour
Solids	: 36.5% (typical)	pH (100%)	: 5.5 - 6.5 (typical)
Odour	: Characteristic	Set Point	: 20 ± 5°C
Viscosity (Carrimed Rheometer @ 20°C : N/A)			

The product had small angle x-ray diffraction peaks characteristic of cubic symmetry and formed a clear microemulsion on dilution with water or warming. The latter gave good even deposition of oil on skin.

**Examples 3 and 4**

The following ingredients were mixed at 60°C and cooled to form ringing gels:

Component	1		2	
	Solids (%)	w/w (%)	Solids (%)	w/w (%)
"EMPIGEN"® CDL30J/35 (22%)	8	36.4	8	36.4
"EMPIGEN"® BB (30%)	8	26.7	8	26.7
"EMPICOL"® LB40 (40%)	4	7.5	3	7.5
"EMPICOL"® CVH (90%)	4	4	---	---
"EMPILAN"® KB2 (100%)	5.5	5.5	6	6
TRIETHANOLAMINE (100%)	1.1	1.1	---	---
CITRIC ACID	1	0.75	0.75	0.75
ETHYLENE DIAMINE				
TETRACETIC ACID	0.05	0.05	0.05	0.05
"KATHON"® CG (100%)	0.05	0.05	0.05	0.05
LIGHT MINERAL (100%)	14	14	20	20
WATER	---	Balance	---	Balance
<b>TOTAL</b>	<b>45.7</b>	<b>100</b>	<b>46.1</b>	<b>100</b>
<b>Appearance</b>	Clear Gel		Clear Gel	

The following ingredients were mixed at 60 °C and cooled to form a clear 'ringing' gel.

### Example 5

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	18	18
"EMPICOL" ® 0251 70 J (70 %)	12	17.2
"EMPICOL" ® CED5 FL (100 %)	5	5
"EMPILAN" ® KBE2 (100 %)	3	3
"EMPILAN" ® KB6 (100 %)	3	3
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

### Example 6

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
HEAVY MINERAL OIL ("KRISTOL" ® M70) (100 %)	18	18
"EMPICOL" ® 0251 70 J (70 %)	10.5	15
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KB2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.5	1.0
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance



**Physical Data**

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for examples 5 and 6**

- i) Charge water and heat to 60°C.
- ii) Add EDTA, sodium benzoate, citric acid and NaOH. Dissolve with stirring.
- iii) Add "EMPICOL" CED5 FL and mix thoroughly.
- iv) Add glycerol.
- v) Add NaCl and disperse with stirring.
- vi) Add "EMPILAN" KBE2 and "EMPILAN" KB6 or "EMPILAN" KB12. Disperse with stirring.
- vii) Add "EMPIGEN" BB.
- viii) Add mineral and disperse with stirring.
- ix) Add "EMPICOL" 0251 70J and disperse with stirring.
- x) Add additional nonionic surfactant to clear (if necessary).
- xi) Cool to 40°C.
- xii) Add evaporated water
- xiii) Adjust pH and offload.

Example 7

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	9	9
DOW CORNING DC 556 SILICONE FLUID (100 %)	9	9
"EMPICOL" 0251 70 J (70 %)	12	17.2
"EMPICOL" CED5 FL (100 %)	5	5
"EMPILAN" KB2 (100 %)	3.5	3.5
"EMPILAN" KB12 (100 %)	3.5	3.5
"EMPIGEN" BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

The formulation forms a microemulsion at 60°C and forms a gel when cooled to ambient temperature.

**Example 8**

<b><u>Component</u></b>	<b><u>Solids (%)</u></b>	<b><u>W/W (%)</u></b>
HEAVY MINERAL OIL ("KRISTOL" ® M70) (100 %)	15	15
"CERAPHYL" ® GA-D (100 %)	5	5
"EMPICOL" 0251 70 J (70 %)	12	17.2
"EMPICOL" CED5 FL (100 %)	5	5
"EMPILAN" KBE2 (100 %)	3.0	3.0
"EMPILAN" KB12 (100 %)	4.5	4.5
"EMPIGEN" BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	4	4
GLYCEROL (100 %)	2	2
SODIUM HYDROXIDE (50 %)	0.4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

A hazy/opaque emulsion is formed at 60°C and cools to form a clear 'ringing' gel at ambient temperature.

**Physical Data**

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for examples 7 and 8**

- i) Charge water and heat to 60°C.
- ii) Add EDTA, sodium benzoate, citric acid and NaOH. Dissolve with stirring.
- iii) Add "EMPICOL" CED5 FL and mix thoroughly.
- iv) Add glycerol.
- v) Add NaCl and disperse with stirring.
- vi) Add "EMPILAN" KBE2 and "EMPILAN" KB12. Disperse with stirring.
- vii) Add "EMPIGEN" BB.
- viii) Blend 50/50 oil phase - oil and cosmetic ingredient. Add to aqueous surfactant solution. Disperse with stirring to form homogeneous emulsion.
- ix) Add "EMPICOL" 0251 70J and disperse.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Adjust pH and offload.

If gel is opaque, re-heat and add additional nonionic surfactant or water.

**Example 9**

<u>Component</u>	<u>Solids (%)</u>	<u>W/W (%)</u>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 810/812S	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	3.5	3.5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Hazy emulsion clears to form a microemulsion on cooling and 'ringing' gel is obtained at ambient temperature.

**Example 10**

<b>Component</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 818 (100 %)	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" ® KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Forms a microemulsion at 60°C and a 'ringing' gel is obtained after cooling.

**Example 11**

<b>Component</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
LIGHT MINERAL OIL ("KRISTOL" ® M14) (100 %)	10	10
"MIGLYOL" ® 840	10	10
"EMPICOL" ® 0251 70 J (70 %)	11	15.7
"EMPICOL" ® CED5 FL (100 %)	6	6
"EMPILAN" KBE2 (100 %)	3.5	3.5
"EMPILAN" ® KB12 (100 %)	5	5
"EMPIGEN" ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0.5	1
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

**Physical Data**

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for examples 9, 10 and 11**

- i) Blend 50/50 oil phase – oil and cosmetic ingredient. Heat to 60°C.
- ii) Add glycerol and stir to disperse.
- iii) Add “EMPILAN” KBE2 and “EMPILAN” KB12. Disperse with stirring.
- iv) Add “EMPICOL” CED5 FL.
- v) Add “EMPIGEN” BB.
- vi) Add “EMPICOL” 0251 70J.
- vii) Add EDTA, citric acid, sodium benzoate and NaCl. Disperse with stirring.
- viii) Add water.
- ix) Add NaOH.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Adjust pH and offload.

**Example 12**

<b>C mponent</b>	<b>Solids (%)</b>	<b>W/W (%)</b>
EMOLLIENT – FATTY ACID ESTER (100 %)	20	20
“EMPICOL” ® 0251 70 J (70 %)	12	17.2
“EMPICOL” ® CED5 FL (100 %)	5	5
“EMPILAN” ® KB6 (100 %)	5	5
“EMPIGEN” ® BB (30 %)	3	10
SODIUM CHLORIDE (100 %)	5	5
GLYCEROL (100 %)	1	1
SODIUM HYDROXIDE (50 %)	0,4	0.8
ETHYLENE DIAMINE TETRACETIC ACID (100 %) Na SALT	0.1	0.1
CITRIC ACID (100 %)	0.2	0.2
SODIUM BENZOATE (100 %)	0.3	0.3
WATER	-	Balance

Clear gels have been prepared using the following fatty acid esters:

Isopropyl laurate (“ESTOL” ® IPL 1505)  
 Isopropyl myristate (“ESTOL” ® IPM 1512)  
 Isopropyl palmitate (“ESTOL” ® IPP 1517)  
 Isopropyl isostearate (“SCHERCOMOL” ® 318)

**Physical Data**

Density @ 20°C : 1.0 +/- 0.1

pH (10 %) : 5.5 +/- 0.5

Appearance: Clear or hazy gel

Odour : Characteristic

Set point (typical): 35 +/- 5°C

Viscosity @ 20°C: N/A

**Method for example 12**

- i) Heat oil phase to 60°C.
- ii) Add “EMPILAN” KB6 and stir to disperse.
- iii) Add glycerol and stir to disperse.
- iv) Add “EMPIGEN” BB.
- v) Add “EMPICOL” CED5 FL.



- vi) Add "EMPICOL" 0251 70J.
- vii) Add EDTA, NaCl, sodium benzoate and citric acid. Stir to disperse.
- viii) Add water.
- ix) Add NaOH.
- x) Cool to 40°C.
- xi) Add evaporated water.
- xii) Check pH (10 %).
- xiii) Adjust pH and offload.

The products in each case exhibited cubic symmetry and formed clear microemulsions or dilution with water or heating. The registered trade marks noted above have the following significance:-

"EMPICOL" CVH is a C<sub>8</sub> alkyl ether carboxylic acid

"EMPICOL" LB40 is a C<sub>8</sub> C<sub>10</sub> alkyl sulphate

"EMPICOL" 0251/70J is a C<sub>12-14</sub> alkyl 3 mole ethoxy sulphate

"EMPICOL" 9758 is a C<sub>10</sub> alkyl sulphate

"EMPICOL" CED 5FL is lauryl 6 mole ethoxy carboxylic acid

"EMPIGEN" BB is a C<sub>12-14</sub> alkyl betaine

"EMPIGEN" CDL is coconut amphoteric acetate

"EMPILAN" KB2 is a C<sub>12-14</sub> alkyl 2 mole ethoxylate

"EMPILAN" KB6 is a C<sub>12-14</sub> alkyl 6 mole ethoxylate

"EMPILAN" KB12 is a C<sub>12-14</sub> alkyl 12 mole ethoxylate

"GLUCAPON" 215CS is a C<sub>8-10</sub> alkyl polyclucoside D.P. 1.5

"KATHON" CG is a proprietary biocide

"DOW CORNING" DC556 is phenyl trimethicone

"CERAPHYL" GA-D is maleated soya bean oil

"MIGLYOL" 810/812S is capric/caprylic triglyceride

"MIGLYOL" is capric/caprylic/linoleic triglyceride

"MIGLYOL" 840 is dipropylene glycol dicaprylate/dicaprate

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CLAIMS

1. A concentrated personal cleansing composition comprising, by weight of the composition, at least 20% water, 10 to 40% total surfactant and 2 to 40% of oil wherein said surfactant comprises (A) an oil soluble surfactant having an HLB of from 2 to 10 in a proportion of from 8:1 to 1:5 based on the weight of oil and (B) a hydrophilic surfactant having an HLB greater than 11, in a weight proportion of from 1:1 to 1:30 based on the weight of (A), said surfactant water and oil being present in proportions adapted to form an  $I_1$  phase having an  $I_1/L_1$  transition temperature greater than 25°C.
2. A composition according to claim 1 wherein the total surfactant has a mean HLB between 10 and 15.
3. A composition according to claim 1 wherein said oil comprises a mineral, fatty ester, glyceride, terpene or silicone oil
4. A composition according to either of claims 1 and 3 wherein the oil comprises at least 16% based on the weight of oil, of a mineral oil.
5. A method for preparing a composition according to claim 1 comprising : (i) forming a mixture (a) of said oil and said oil soluble surfactant; (ii) mixing said mixture (a) with a mixture (b) of said water soluble surfactant and sufficient water to form a lamellar phase with said water soluble surfactant; (iii) maintaining said mixture of (a) and (b) above the  $I_1/L_1$  transition temperature of said composition while diluting said mixture of (a) and (b) with water to form said composition; and (iv) cooling said composition below the  $I_1/L_1$  transition temperature.

TRANSMITTAL LETTER TO THE UNITED STATES  
DESIGNATED/ELECTED OFFICE (DO/EO/US)  
CONCERNING A FILING UNDER 35 U.S.C. 371

01795/HG

APPLICATION NO. (If known, see 37 CFR 1.5)

INTERNATIONAL APPLICATION NO.

PCT/EP00/05341

INTERNATIONAL FILING DATE

JUNE 9, 2000

PRIORITY DATE CLAIMED

JUNE 10, 1999

TITLE OF INVENTION

PERSONAL CARE FORMULATIONS

APPLICANT(S) FOR DO/EO/US Kevan HATCHMAN, Elvin LUKENBACH, Laura MCCULLOCH and  
Benjamin WIEGAND

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a FIRST submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☒ The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
- a. ☒ is attached hereto (required only if not communicated by the International Bureau). (As WO 00/76460 A2)
- b. ☒ has been communicated by the International Bureau.
- c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
- a. ☐ is attached hereto.

FRISHAUF, HOLTZ, GOODMAN, LANGER &  
CHICK, P.C.ATTORNEYS-AT-LAW  
767 THIRD AVENUE, 25TH FLOOR  
NEW YORK, NY 10017-2023

84229

1-108/210

December 7, 2001 DATE

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THIS CHECK IS DELIVERED IN CONNECTION WITH THE FOLLOWING ACCOUNT(S)

01795/HG - HATCHMAN et al.	
New PCT Application "PERSONAL	
CARE FORMULATIONS"	
Filing Fee	

FRISHAUF, HOLTZ, GOODMAN  
LANGER & CHICK, P.C.

⑈084229⑈ ⑆021001088⑆ 615001939⑈

12. ☐ A FIRST preliminary amendment.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☒ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.321 - 1.325.
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information:

- (i) Copy of WO 00/76460 A2
- (ii) PCT/ISA/210 (Search Report)
- (iii) PCT/IB/304 (Priority Document Sent)
- (iv) PCT/IB/308 (Appln. sent to U.S.)
- (v) REQUEST FOR PUBLICATION OF ASSIGNMENT INFORMATION

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No.: EL 874 117 723 US  
Date of Deposit: December 7, 2001I hereby certify that this paper is being  
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date indicated above and is addressed to  
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